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CONTENTS.

The Restriction of Insect Infestation to the Periphery of Bulk Wheat.

*By Frank Wilson.**

Summary.

Insect infestation in Australian bulk-wheat storages is restricted to a narrow zone at the periphery of the wheat mound. This restriction develops gradually during the history of an infestation, and is caused by the heat generated by the insects, the loss of moisture resulting from the rise in wheat temperature, and the consequent development of a combination of temperatures and moisture contents within the grain mound which is lethal to the insects. The temperature of the interior lethal zone is maintained by insect activity in the peripheral zone, where higher wheat moisture contents permit insect development at temperatures which are as high as, or higher than, those occurring in the lethal zone.

1. Introduction.

During the present war, vast quantities of wheat have been stored in Western Australia and Victoria in bulk storages of different types, in which the wheat is held in a single mound. The mound may be relatively small, as in Western Australian "temporary bulkheads" or "bins," or may be very large, as in long-term storage "depots" of either State, the capacity of a single depot shed being sometimes as great as ten million bushels. All these types of bulk storages, whatever their capacity, have the characteristic that appreciable insect damage is restricted to the periphery of the wheat mound, that is, to a thin layer of wheat at the surface, the walls, and the floor. Quite commonly the damage occurring near the floor is negligible.

This phenomenon is of theoretical interest to the entomologist and of considerable practical importance to those responsible for the safety of Australian wheat stocks, and the underlying causes of the phenomenon have been investigated.

In this article, a brief account will be given of the factors which bring about a restriction of insect infestation to the periphery of bulk wheat, but the data on which the conclusions are based will not be presented. These, together with an account of other aspects of insect infestation in bulk wheat, will be published later.

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2. The Development of Lethal Zones in Bulk Wheat.

Let us consider the example of a block of wheat at the centre of a much larger wheat mass, the whole having, initially, a certain uniform moisture content and a temperature just above the minimum necessary for the development of the insect pests. If this central wheat block becomes infested, insects can breed at any point and will spread throughout it. As a result of insect activity the temperature of the central block will rise, for the heat conductivity of wheat is low and so the dissipation of metabolic heat will be very slow. Observations have shown that this rise in temperature continues until it closely approaches the maximum at which reproduction is possible. If there were no other complicating factor, the density of the insects would reach a state of balance in which heat is produced by the insects at the rate it is dissipated, the temperature being maintained at a level which permits just sufficient reproduction to make up for the natural mortality. Because of the slowness of heat dissipation, the density of the insects would be maintained at a low level.

However, the rise in temperature in the central block increases the vapour pressure and lowers the relative humidity of the atmosphere in the inter-grain spaces of the block. This causes the moisture content of the grain to fall and moisture to pass from the heated block into the surrounding, uninfested and cooler wheat mass. Both heat loss and water loss can only take place through the periphery of the central block, and temperature and moisture content gradients will develop, the temperature falling and the moisture content rising towards the periphery.

This will produce a gradient in insect density, for towards the periphery of the infestation the temperature will be lower and the moisture content higher than at its centre, and conditions will be more favourable for reproduction the greater the distance from the centre. The density gradient will become steeper as the centre continues to lose moisture, and it has been observed that eventually the moisture content in this zone becomes too low for insect reproduction at the existing temperature. From this time the temperature in the central lethal zone will be maintained by the existence around it of a zone of equal temperature in which the insects are able to reproduce because a higher moisture content exists.

This process will continue, the size of the zone possessing a temperature—moisture-content complex which is lethal, gradually enlarging with the passage of time. We have been considering here the changes consequent upon infestation in a central block of wheat lying within a larger wheat mass. This artificial construction was adopted in order to eliminate from consideration important factors which come into operation only at the exterior of a wheat mound. These we may now consider.

The exposed face of a wheat mound is directly subject to the major influences of the atmosphere above it on its temperature and moisture content. But a wheat mass is highly resistant to changes in temperature and moisture content. For example, an annual fluctuation in moisture content of 5 per cent. (9 to 14 per cent.) at the surface of

bulk wheat is reduced to practically zero at a depth of two or three feet. Similarly, an annual fluctuation in temperature of about 55°C. at the surface, is reduced to some 18°C. at a depth of one foot, and 4°C. at nine feet. Atmospheric influences, therefore, decrease markedly in effect with depth, but they have an extremely potent influence on the wheat near the surface of the mound.

Considering first its effect upon the temperature of infested wheat, the atmosphere normally cools the wheat considerably. Insect infestation tends to bring bulk depot wheat to a maximum of 42°C., and a very steep temperature gradient is to be found in the top foot of infested wheat. It is true that in the warmer hours of a hot summer day a very shallow layer of wheat at the surface may be heated to a temperature of 55°C., but, even in summer, the mean daily temperature of the depot atmosphere is well below 42°C. and maintains a steep gradient through the top foot of wheat.

As explained above, an infestation beginning at some depth in the wheat mound will gradually be driven towards the periphery by the development of lethal physical conditions within the mound. When the infestation is eventually forced to the surface, atmospheric influences will greatly affect the physical environment in the shallow zone occupied by the insects. The wheat surface temperature is largely controlled by the atmosphere, irrespective of the underlying wheat temperature, and a steep temperature gradient will occur in the surface layer of wheat. Consequently, in this zone the great heat loss occurring along the steep temperature gradient will permit a high insect density, for this density everywhere is balanced with the maintenance of a given temperature for the existing moisture content. Additionally, the surface zone with its steep temperature gradient will contain relatively cool wheat near the surface, despite the dense insect infestation, and will maintain a higher moisture content than the lower, heated wheat, which is yielding moisture. (There is some evidence that convection currents as well as the greater vapour pressures induced by the high temperatures cause an upward movement of moisture.) Further, the activities of the dense insect populations liberate moisture, and these combined influences produce high moisture contents in the wheat near the surface. This raises the maximum temperature at which the insects can reproduce in this zone.

It is clear from this that in mature surface infestations the insect densities must be greater in winter than in summer, for in winter there will be a much greater heat loss from the surface of the mound, so permitting a greater insect density. Also, in summer, the wheat near the surface loses moisture rapidly to the atmosphere and the reduced moisture content lowers the maximum temperature for insect reproduction and, consequently, lowers the population density.

At parts of the wheat mound periphery other than the surface the same influences operate, but with different intensity. The retaining walls or bulkheads in bulk storages are either of wood or corrugated iron. Where these are of wood, they provide some insulation against heat and moisture transfer, and insect infestation is limited thereby. Where there is a corrugated iron bulkhead this is no impediment to heat loss and a higher insect density is consequently permitted. Further, the extreme diurnal temperature fluctuations to which the iron is

subjected cause the condensation of moisture on its interior surface
and a great increase in the moisture content of adjacent wheat results.
This raises the maximum temperature at which the insects can breed,
and consequently raises the insect density in this zone.

Insect infestation at the floor of a bulk storage is markedly limited
by the relatively low rate at which heat is lost to the soil beneath.
It is this factor which makes floor infestations of little importance
compared with those occurring at the surface and at the walls.   A
floor infestation is normally of negligible importance if the floor is
soundly constructed of sheet-iron or concrete.   When the floor is com-
posed merely of a layer of bitumen, however, there is often a con-
siderable increase in moisture content and the infestation is much
heavier.

In examining the processes which bring about the restriction of
infestation to the periphery of bulk wheat, the example was taken
of an infestation beginning in the centre of the mound.   However,
in a wheat depot insect infestations begin most frequently at, or close
to the surface.   There are two reasons for this.   First, the great mass
of the wheat is generally low in moisture content and temperature
when it is placed in store, and it is only near the surface that these
(at certain times of the year) are considerably increased by atmospheric
influences.   Secondly, it is only at the surface that insect dispersal can
take place readily.   Consequently, it commonly happens that an infesta-
tion begins in the zone to which the insects will eventually be
restricted, and the lower levels of the wheat will be heated from the
inception of infestation by the downward passage of heat from the over-
lying infestation.   This does not, of course, prevent the insects from
penetrating a considerable distance into the wheat mound, but must have
the effect of reducing the amount of insect damage which occurs at the
lower levels before the changes in physical conditions render reproduc-
tion impossible there.

3. The Bulk Storage of Wheat of High Moisture Content.

So far during this war the wheat placed in bulk storages has been
of a moisture content generally not above 10 per cent.   Whether insect
infestation would still be restricted to the peripheral zone if wheat of
high moisture content were placed in bulk storage is a question of
considerable practical importance.

If wheat of, say, 12 or 13 per cent. moisture content were stored in
a bulk depot, insect infestation would develop in a similar manner to
that occurring in wheat of low moisture content.   It would differ from
this, however, in the relative importance of the insects involved.   (In
particular, rice weevil would be far more numerous.)   Nevertheless,
the same fundamental processes would take place.   The temperature
would approach the maximum at which reproduction could take place;
moisture would be lost by the heated wheat; lethal zones would develop;
and the infestation would become restricted to the periphery.   But
because the higher moisture content would permit reproduction at
higher temperatures, the damage caused by the insects would be some-
what greater before these higher temperatures were reached.   The
infestation would eventually be restricted to the surface, and the insect

density determined by the rate of heat loss at the periphery. However, because the temperature at which reproduction is possible would be higher, the temperature gradient would occur through a somewhat greater depth of wheat and insect damage would be consequently increased.

More important results of the storage of wheat of high moisture content would be, firstly, the possibility of the development of bin scald, and, secondly, the virtual impossibility of controlling the infestations as they arose. Methods of fumigating surface infestations in bulk depots have been developed, and their application has kept the insect pests under control. With wheat of high moisture content, however, the physical conditions would so favour insect development that these methods would be unable to cope with the numerous outbreaks of insects. Probably no methods that would be considered commercially practicable could be devised to meet such a situation, and it would probably be necessary to permit the wheat mound to heat so that the pests became restricted to the peripheral zone. The damage caused could be minimized by taking measures to reduce so far as possible the loss of heat from the wheat mound.

4. Acknowledgments.

Mineral Chlorination Studies.

I. Production of Titanium Tetrachloride from Australian Rutile Sand.

By F. K. McTaggart, M.Sc.*

Summary.

A process, which has been operated on pilot-plant scale, for the production of titanium tetrachloride from the rutile occurring in the zircon-rutile sands of the north-east coast of Australia is described. This process involves briquetting the unground, or suitably ground, sand with carbon, coal, &c., and a binder such as tar, and thereafter chlorinating the briquettes in a furnace. It appears that, to date, this type of reaction has been carried out with little attempt to determine optimum working conditions. Because of the wartime demand for titanium tetrachloride and the subsequent production of this material in Australia, it was thought desirable to determine these conditions by a study of such factors as the variation of reaction rate with temperature, particle size, type and composition of briquette, &c., and an investigation into materials suitable for the construction of large-scale plant and the difficulties likely to be encountered in the operation of such a plant.

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1. Introduction.

One of the major aims of the Minerals Utilization Section of the Division of Industrial Chemistry is to develop methods for the exploitation of minerals with which Australia is comparatively well endowed. One of Australia's potentially valuable mineral resources is the beach sands of her north-eastern coastline. These beach sands are natural concentrates of the minerals zircon, rutile, ilmenite, and monazite. Adequate utilization of rutile will entail long studies of the chemistry of titanium compounds. In the first instance titanium tetrachloride has been investigated because it has an important wartime significance.

It is well known that titanium tetrachloride can replace stannic chloride in a number of applications, and with the rapidly developing shortage of tin it was decided in 1942 to commence investigations on the production of titanium tetrachloride from rutile as an alternative material.

It had been proposed to produce titanium tetrachloride from imported titanium white pigment, but the researches described in this publication demonstrated that this would be a retrograde economic procedure. They also demonstrated that the effect of grinding the rutile was less than might have been anticipated.

There are numerous references (1) to the reaction between various forms of titanium dioxide and carbon and chlorine, and it is known that this reaction has been used commercially. Operating details of commercial plants have not been published, and such information as it has been possible to obtain suggests that much has still to be learned concerning the reaction. The present account describes an attempt to determine optimum conditions of operation, using as raw material rutile from Australian beach sands.

Irrespective of the form of the titanium dioxide used the reaction is:—

Equation .. .. .. $TiO_2 + 2C + 2Cl_2 = TiCl_4 + 2CO$ . . . (I)
Molecular weight ratio .. $79 \cdot 9 + 24 + 142 = 189 \cdot 9 + 56$
Heats of formation 25°C. (ΔH) $217 \cdot 4$ $183 \cdot 5$ $26 \cdot 4$ K. cals.
Heat given out .. .. $18 \cdot 9$ K. cals. per gram molecule of TiO_2
Free energies at 25°C. (ΔF)* $180 \cdot 5$ K. cals. $165 \cdot 1$ $12 \cdot 9$ K. cals.
Difference in free energy .. $10 \cdot 4$ K. cals. per gram molecule of TiO_2

Sufficient thermodynamical data concerning titanium dioxide and titanium tetrachloride are not available to permit the accurate calculation of the quantity of heat given out at the temperature of reaction (700°C.), but it would appear that the reaction is slightly exothermic.

The theoretical proportion of titanium dioxide to carbon, $79 \cdot 9$ to 24, is approximately $3:1$, and this ratio has been used throughout the experimental work. Analyses of residues (see later) show that this ratio is satisfactory.

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* Calculated from the equation $\Delta F = \Delta H - TdS$, using values of dS for rutile ($12 \cdot 4$), titanium tetrachloride (liquid, 60) and carbon monoxide (47) given in the United States Department of the Interior Bulletin (No. 434), entitled " Contributions to the Data on Theoretical Metallurgy. IX. The Entropies of Inorganic Substances ".

Initially it was realized that there might be a tendency for the reaction to proceed according to the equation:—

$$TiO_2 + C + 2Cl_2 = TiCl_4 + CO_2 \quad .. \qquad .. \quad (II)$$
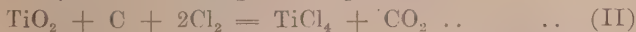
but as considerable evidence was found to prove that this is not so, it is not considered further. This evidence is discussed in a later section.

2. Raw Materials.

Raw materials used were:—

(i) *Rutile.*—The rutile was obtained as a commercially separated fraction produced from the zircon-rutile sands at Byron Bay, New South Wales. This sand consists chiefly of the minerals zircon, rutile, ilmenite, with subordinate amounts of other heavy minerals (2). The initial concentration of these minerals is the result of tidal and wave action on the beach sands of the locality. The component minerals are separated on an industrial scale by a combination of processes involving selective flotation, gravity concentration, and magnetic separation. The lastnamed process is used to separate the rutile from the mineral fraction consisting of ilmenite and rutile—the rutile being non-magnetic. A product of high purity is obtained.

Two sizes of rutile were used for the laboratory and pilot plant testing:—

(*a*) Unground sand as obtained from the magnetic separator; this was substantially 100 mesh to 120 mesh. The particles were smooth, black and shiny, and contained by analysis 2·1 per cent. of Fe_2O_3, 4·2 per cent. of SiO_2 and 93·0 per cent. TiO_2.

(*b*) Ground sand having the following screen analysis:—

> All passed through 160 mesh screen.
> 15·6 per cent. between 160-200 mesh.
> 35 per cent. between 200-300 mesh.
> 49·4 per cent. passed through 300 mesh.
> The sand contained 0·8 per cent. Fe_2O_3, 1·8 per cent. SiO_2, and 97·1 per cent. TiO_2.

Throughout the following discussion these two samples will be referred to as "unground" and "ground" rutile respectively.

(ii) *Charcoal.*—High-grade activated charcoal as used for decolourizing purposes was employed during laboratory scale determinations of the effect of particle size of rutile on reaction rate (1st series). Experiment showed, however, that high-class gas-producer charcoal ground to approximately 200 mesh was just as satisfactory, and a quantity of this was used for pilot plant production of titanium tetrachloride prior to the use of coal.

(iii) *Coal.*—Black bituminous coal was crushed in a small jaw crusher and ground in a Braun mill until it had the following screen analysis:—

> 10 per cent. retained on 50 mesh screen.
> 41·5 per cent. retained on 100 mesh screen.
> 29·5 per cent. retained on 150 mesh screen.
> 12·0 per cent. retained on 200 mesh screen.
> 6·0 per cent. passed by 200 mesh screen.

Ash analysis of the coal resulted in a total ash content of $11 \cdot 5$ per cent. made up by—

SiO_2	..	..	..	..	$7 \cdot 6$ per cent.
Fe_2O_3	..	..	..	..	$0 \cdot 5$ per cent.
Other R_2O_3		..	..	..	$2 \cdot 6$ per cent.
Undetermined alkalies, &c.		..	..	..	$0 \cdot 8$ per cent.

$11 \cdot 5$ per cent.

(iv) *Tar.*—Three types of tar were investigated for bonding of briquettes :—

(*a*) Crude horizontal still tar. This is usually contaminated with water, an undesirable feature when later heating is necessary, but the sample used had been freed from water by long standing. It was a very thin black tar with excellent bonding properties, but this material was in short supply owing to the increasing tendency by gas companies to use vertical retorts. Attention was therefore turned to vertical still tars.

(*b*) A fairly heavy fraction of redistilled vertical still tar. This was a pitch-like material, and was unsuitable firstly because it had to be heated before mixing, and secondly because of its change in viscosity with increase in temperature (see below).

As (*c*) below became the final choice, further details concerning (*a*) and (*b*) are omitted.

(*c*) A light fraction of redistilled vertical still tar which had the following physical and chemical characteristics* :—

Specific gravity (15°C.)	..	..	$1 \cdot 06$	
Free carbon	..	..	5 per cent. by weight	
Water	..	..	..	Nil

Distillation range—

0–170°C.	..	..	..	Nil
170–230°C.	..	..	..	$8 \cdot 6$ per cent. (by volume)
230–300°C.	..	..	..	28 per cent. (by volume)
Total oils to 300°C.	..	..	..	$36 \cdot 6$ per cent.(by volume)
Pitch content	..	..	..	63 per cent. (by weight)

This tar, which was thin and dark-brown in colour, was found suitable for briquetting purposes.

3. Experimental.

(i) *Briquetting.*

The term " briquetting " is used to designate the formation of hard aggregates of the rutile-carbon mixture independent of the means employed to accomplish this. Thus the process may be carried out by machine or press, or simply by rolling into balls by hand the final aim was, in every case, the production of aggregates having sufficient

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*We are indebted to Messrs. James Forbes (Tar Distillers), Melbourne, for this analysis, which is stated in the conventional tar manufacturers' way. This specification is very close to what is known in Australia as No. 1 vertical tar, and could be described as " No 1 vertical tar plus 3 per cent. of light oils."

strength to withstand handling, shovelling, &c., and to resist breakdown and crumbling in the furnace due to their own weight. Sufficient porosity to allow adequate penetration was also a necessary characteristic.

The variables in a briquetting process are: the type of binder, the quantity of binder, the pressure employed in moulding, and the particle size or sizes of the moulded material. Considerable work was carried out using rutile and charcoal in the previously mentioned ratio of 3:1, bonded with such bonding agents as 5, 10, and 15 per cent. starch solutions, 10 and 20 per cent. sugar solutions, solutions of dextrose, dextrine, molasses, &c. Various pressures from 200 to 6,000 lb. per square inch were tried, and finally excellent briquettes were produced, for example, with 15 per cent. starch solution at 4,000 lb. per square inch, and with 75 per cent. molasses solution at 200 lb. per square inch. However, apart from one series of laboratory scale experiments, these were not used because we were given details of a process involving no pressure, which could be carried out on a large scale with simple equpiment. As this method became the final choice, a discussion of the other methods mentioned above is omitted. It consisted of mixing three parts by weight of rutile with one part of ground coal and then mixing with crude tar, of which about two parts by weight were necessary to give a thick pasty mass. This mass was then spread out on an iron tray making a layer from $\frac{1}{2}$ to $\frac{3}{4}$ in. in thickness. The tray was heated strongly by means of burners or a fire underneath, and when gases were evolved from the tarry mass, the latter was set alight also. Fierce burning from the surface then continued for about half an hour, after which it subsided and finally ceased. The fire underneath the tray was then put out and the tray and contents were allowed to cool. The mass when cold was found to consist of a layer of hard coke-like material, very porous and quite strong. It was broken into suitably sized lumps and ignited out of contact with the air to 600°-700°C. to drive off the remainder of the volatile material from the coal and tar and was then ready for chlorination. The optimum proportions of rutile, coal and tar are 12:4:7 parts by weight, and analyses of the porous masses resulting from this mixture showed that the ratio of rutile to carbon always lay between 2·8 and 3·2 to 1.

In calculating the rutile-carbon ratio of the briquettes, after ignition out of contact with air, it should be noted that if the carbon is being determined by loss of weight following complete oxidation, the residue contains coal ash as well as rutile. In the present case, of the total residue, the amount of ash was about 4 per cent., which was allowed for. A further correction could have been applied, because about one-third of the ash consumed chlorine in the furnace with a consequent utilization of a small amount of carbon, but this was neglected.

Provided that the ratio of rutile to carbon did not fall below 3:1, there was a small excess of carbon recoverable in the residue from chlorination.

The tar used for mixing with the rutile-coal must be thin. If not, it is too difficult to mix without heating, but an even less desirable feature of a thick tar is its change in viscosity when the paste is being " burnt off." It becomes so fluid that it tends to drain away from the solid materials, leaving them with insufficient binding agent. A thin tar on

the other hand suffers little change in viscosity, and the paste is practically unaltered in consistency when heated. However, the tar should not contain too much oil, as this has little or no binding properties—its fluidity should be a natural one, and not artificially brought about by the addition of petroleum, &c.

(ii) *Laboratory Scale Chlorinations.*

(*a*) *Description of apparatus* (Fig. 1). The laboratory furnace consisted of a vertical silica tube (A), $1\frac{1}{4}$ in. internal diameter and 20 in. long, wound over the central 18 in. with 26 gauge nichrome wire over which was wrapped alternate layers of asbestos sheeting and cord to a thickness of $\frac{3}{4}$ in. (B). At the bottom, an asbestos stopper (D) held the chlorine inlet (C) and a thermocouple sheath (E) running the full length of the furnace. The charge, which usually consisted of 100 g. of $\frac{3}{8}$ in. briquettes, rested on a bed of $\frac{1}{4}$ in. silica chips (F). This bed of chips was 2 in. in depth, and held the lowest portion of the charge up in the hot zone of the furnace as well as providing a preheating zone for the chlorine and preventing the inlet tube becoming blocked with dust from the briquettes. Before entering the furnace the chlorine passed through a sulphuric acid bubbler (G) and a flowmeter of the orifice type (H) calibrated for rates up to 400 cc./min. A Liebig condenser (I) was attached to the furnace by means of a ground glass jointpiece (J); it condensed all the titanium tetrachloride produced in the furnace, even at the fastest rate. Cooling by tap water was
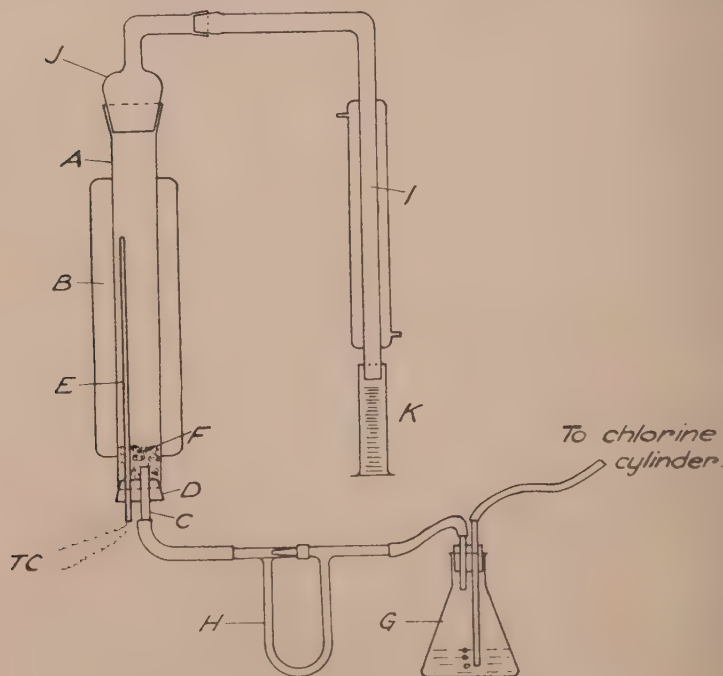

FIG. 1.—Diagram of laboratory chlorination apparatus.

quite effective. The receiver (K) was usually a graduated glass cylinder
packed in ice. Fuming of the titanium tetrachloride could be almost
entirely eliminated by having the end of the condenser reaching well
into the receiver.

(b) *Variation of reaction rate with temperature.* The reaction for
all types of briquettes, independent of grain size or binder, was found
to commence at 450°C. This may readily be verified by placing a
sample of briquettes (50-100 g.) in the furnace, and increasing the tem-
perature in steps of 10°C. while passing chlorine. The green colour of
the unused chlorine gas is clearly visible in the condenser until 450°C.
is reached, when white fumes appear showing that titanium tetra-
chloride is being formed. It has been reported (3) that the reaction
is catalysed by rare earth chlorides and that in the presence of a small
proportion of these, the reaction proceeds at 100 to 200°C. less than
the above. The effect of additions of 1 per cent. and 5 per cent. rare
earth chlorides was tried. In neither case was any difference in the
reaction rate detected, and the white fumes first appeared at 450°C. as
before.

For determining the variation in reaction rate with temperature, a
series of tests was made using porous type briquettes of $\frac{3}{4}$-in. mesh made
from ground rutile; 100 g. charges were used at a chlorine rate of
200 cc./min., which, from previous experiments, was known to be slightly
in excess of the amount the charge could take up. Thus all conditions
were held constant except the temperature. Tests were made at 450°,
500°, 600°, 800°, and 1,000°C. Fig. 2, in which temperature is
plotted against yield of TiCl$_4$ (reduced to mean value in cc. per 10
min.) shows that the reaction rate increases rapidly up to 600°C., but
from then on scarcely alters. This indicates that a temperature of
about 700°C. is adequate for the chlorination; this temperature has
been used in the pilot plant work.
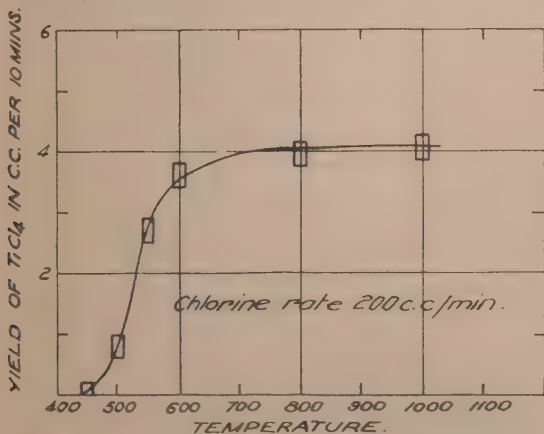
Fig. 2.—Influence of temperature on reaction rate.

(c) *Variation of reaction rate with particle size.* Two series of
experiments were made to determine the influence of particle size. In
the first series starch-bonded, pressure-moulded briquettes were used,
and in the second tar-bonded, porous briquettes. Contrary to expecta-
tion, results showed that the pressure-moulded test aggregates were

somewhat better than the porous type, although they have not the
characteristic vesicular appearance of the latter. This obvious
" porosity " may not be the true porosity at all. In other words,
although the starch briquettes are denser and apparently non-porous
from the point of view of heterogeneous reaction they may exhibit a
greater reactive surface to the gas than the obviously vesicular type.

In the determination of the effect of particle size, all other factors
had to be kept constant and conditions were standardized in the follow-
ing manner :—

First series.—Temperature held at 600°C., charges : 100 g. of pressed
(4,000 lb. per square inch) briquettes of rutile and carbon bonded by
10 per cent. starch solution. The determination was complicated by the
necessity of determining whether or not sufficient chlorine was entering
to satisfy the charge completely. The quantity necessary would be con-
siderably greater for a fast reacting charge than for a slow one, so that
for each sized particle three chlorinations were made, using chlorine
rates of 100 cc., 200 cc., and 250 cc. per minute. Hence each particle
size is represented by three " points " in Fig. 3. Four sizes of rutile were
tested as shown in the figure. Points are also included for titanium
white pigment. The " points " are self-explanatory and show also by
their size the magnitude of the experimental errors in making the
observations of yield and chlorine rate. The straight line represents
the theoretical yield.
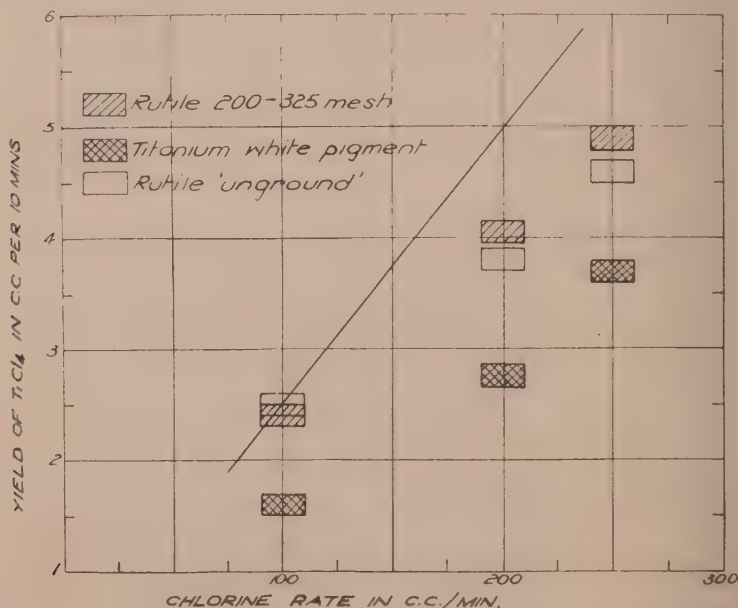
Fig. 3a.—Reactivity of first series (Starch bonded) briquettes shown by
use of different chlorine rates.

The first comment that may be made concerns the poor reactivity
of the titanium white pigment compared with the various sizes of rutile.
It was thought that the finer the particle size the faster the reaction rate

would be, and hence the white pigment was expected to prove superior to all the others. On the contrary, it is definitely inferior, and no satisfactory explanation of this can be given at this stage, because analysis has shown that the pigment contains little impurity, while previous chlorination experience has shown that impurities such as might be found in it have practically no effect on rate of chlorination. It may be due to a difference in crystalline structure between the rutile and the TiO_2 as titanium white, or it may be due to the tighter packing of the smaller particles in the briquette with subsequent reduction of porosity. The same effect has been noticed in the second series using vesicular briquettes.
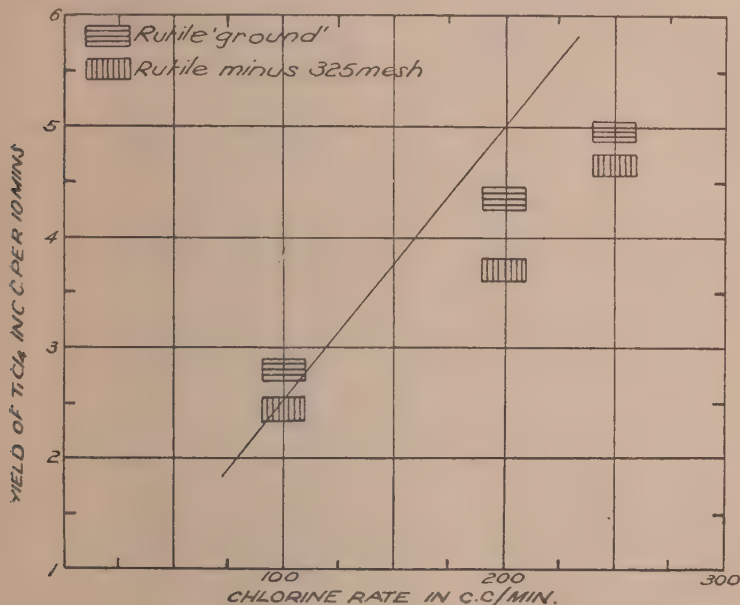
Fig. 3B.—Reactivity of first series (Starch bonded) briquettes shown by use of different chlorine rates.

For the low chlorine rate (100 cc./min.) it is obvious that for all sizes of particles (except the white pigment), the charge was completely absorbing the chlorine and yielding, within the limits of the experimental errors, theoretical quantities of tetrachloride. For the 200 cc. rate the efficiency falls off, but there is little difference between the points. At the highest rate (250 cc./min.) there is a further increase in rate of production of the tetrachloride. This must be explained by the more rapid sweeping away of the products of reaction by the excess chlorine. The points again lie close together, even closer than those for the lower rates. The ground rutile appears to be the best at all rates. The fact that it consists of a mixture of particle sizes perhaps causes this but it is doubtful if any difference could be inferred between the various sizes of rutile that would be important from the point of view of practical application.

Second series (Fig. 4). This series was carried out in a similar manner to the first series, but the briquettes were of the tar-bonded,

porous type. All other factors were kept constant. However, only the ground and unground rutile and the titanium white were tested. Once again the latter proved noticeably inferior to the rutile, while there was no significant difference between the ground and unground rutile. This encouraged the belief that the unground sand could be used in full-scale production of titanium tetrachloride, a belief substantiated by pilot plant experiments (see later).
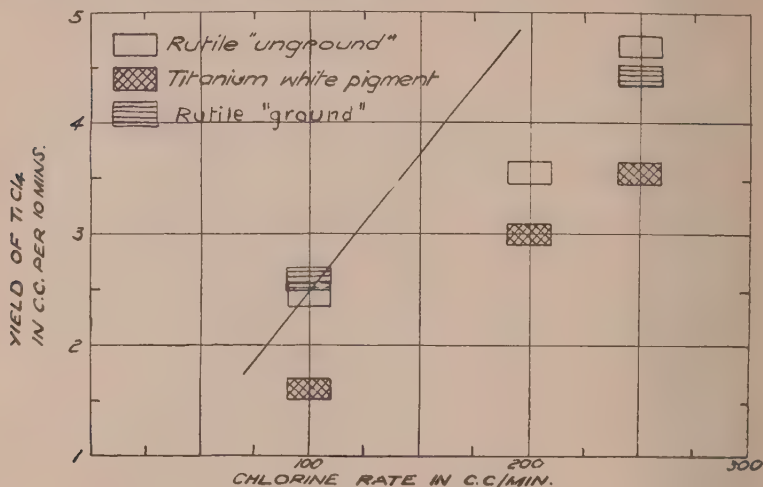
FIG. 4.—Reactivity of second series (Tar bonded, porous) briquettes shown by use of different chlorine rates.

A comparison of the two sets of points shows that the porous type of briquette is not so effective as the pressed and starch-bonded variety, but the difference is small, and in view of the great ease of production of the former it was chosen for larger-scale work.

(d) *Extent of reaction and efficiency.* Laboratory scale chlorinations showed that an efficiency of 83-90 per cent. on chlorine could be easily obtained, but little or no attempt was made to obtain accurate figures, as it was thought that operation of the pilot plant would yield more reliable data. Usually from two-thirds to three-quarters of the total charge was used before passage of the gas was stopped. There appeared no reason why the reaction should not be carried a little further, although in such a case the rate of entry of chlorine would have to be progressively reduced.

Analyses of the residues from experimental chlorinations such as are described above, provided proof that little carbon dioxide was produced during the reaction. Certainly the reaction did not proceed according to the equation—

$$TiO_2 + C + 2Cl_2 \longrightarrow TiCl_4 + CO_2$$
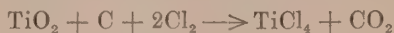
Molec. wts. $79 \cdot 9 + 12 + 142$

because if this had been so half the carbon in the charges would have been recovered unreacted. Figures showed rather that within close

limits the amount of carbon necessary to form carbon monoxide had been used. Further evidence was obtained from a consideration of the equilibrium—

$$C + CO_2 \rightleftharpoons 2CO$$

at the reaction temperature 700° C. Provided equilibrium could be obtained in the furnace, an unlikely occurrence when the gases were continually swept away, the ratio would have been $CO : CO_2 = 62 : 38$ (Stansfield, 4). Thus, only 38 per cent of carbon dioxide would have been present.

(e) *Purification of titanium tetrachloride.* The crude liquid obtained from the furnace contained ferric chloride in solution and in suspension, traces of suspended carbon dust, and dissolved silicon tetrachloride and chlorine; it was yellow to orange in colour. Titanium tetrachloride from the chlorination of titanium white was considerably paler in colour—usually a pale lemon—and it contained practically no sediment or suspension of ferric chloride. However, it always contained free chlorine and sometimes traces of carbon dust. Although the product from the chlorination of rutile does contain more impurity than that from titanium pigment, for many purposes it should prove just as effective, particularly for the formation of smoke screens. It may be purified by the simple purification methods to be described later, and the additional processing required over that necessary for the product from titanium white would be small.

The literature contains a considerable number of references to methods of purification, including refluxing (or standing or agitating) over mercury (5), copper (6), reduced iron (7), sodium (8), potassium (9), sodium amalgam (10), &c. The yellow colour has been stated to be due to traces of vanadium oxychloride (11), which can be removed by distillation over copper. All the above methods have been tried but, with the exception of that employing copper, have proved unsatisfactory. If the titanium tetrachloride is refluxed over copper turnings or foil until colourless and then distilled from the copper, a water white product of high purity results. The copper, which effectively removes the ferric chloride, free chlorine, &c., becomes black, but may be cleaned by immersion in dilute nitric acid and reused a number of times. Sufficient copper must be present, otherwise it becomes inactive and even continued refluxing will fail to clear the tetrachloride. Approximately 3 litres of crude tetrachloride may be purified with 200 grams of copper foil, but it is preferable to allow the ferric chloride to settle and to decant the clear crude liquid before treating with copper. An alternative is to distil the titanium tetrachloride before the copper treatment. Considerably less copper is needed if one of these pretreatment processes is employed. Analyses of titanium tetrachloride at various stages of purification, together with an account of the final method adopted in purifying, will be found in a later section.

Silicon tetrachloride. Although several accounts are to be found in the literature of the preparation of silicon tetrachloride by chlorination of a mixture of silica and carbon at a bright red heat (12), we have found that at 700° C. there is very little attack of the silica content of the briquettes. The maximum amount of silicon tetrachloride found

in the titanium tetrachloride has been only 0·5 per cent., the bulk of the silica always appearing in the furnace residues. In order to determine whether silicon tetrachloride is formed at slightly higher temperatures, a mixture of finely ground silica sand and carbon was chlorinated at 1,000 C.—the maximum attainable with the laboratory furnace. After six hours only 10 per cent. of the silica had disappeared from the charge. Apparently higher temperatures must be reached before this reaction proceeds at an appreciable rate.

4. Pilot Plant Chlorinations.

(i) *Description of the Plant.*

The furnace proper (Figs. 5 and 8) consisted of a tube 4 ft. 6 in. long, 8 in. internal diameter, and 10 in. external diameter, made from pressed silicon carbide (A). This tube was built up from three sections cemented together with a cement containing powdered silicon carbide.*
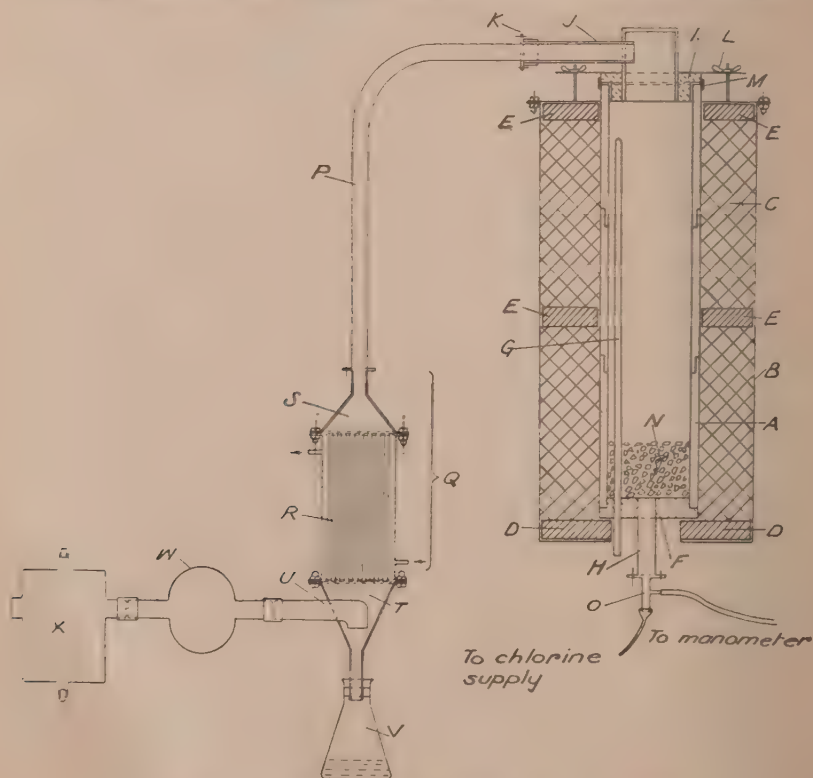
FIG. 5.—Diagram of pilot plant, chlorination furnace, condenser, etc.

*Fabrication of the furnace and end plates was by Nonporite Ltd., Burwood-road, Hawthorn, Melbourne—the trade name for the material being "Carbolox." The jointing cement used was also a product of this firm and is known as "Refractite No. 1."

Preliminary tests, which included treatment at 800°C. for 96 hours in an atmosphere of chlorine, were made on the silicon carbide. The data collected led to the belief that this material would be suitable for the pilot plant, a belief that was substantiated during the subsequent use of the plant. At the time of writing the furnace had seen about 300 hours of operation and there was no sign of attack or deterioration.

The furnace tube was coated with an ⅛-in. layer of alundum cement over which was wound the heating coil of 21-gauge nichrome wire. This winding was in three sections and was operated from the 400-volt supply (see Fig. 6). It was covered by a further ¼-in. layer of alundum. The furnace was surrounded by an outer iron cylinder 2 ft. 2 in. in diameter (B), the thermal insulation consisting of a loose packing of expanded vermiculite granules (C). At the lower end the furnace rested on a circular fireclay brick (D), and was held concentrically with the outer cylinder by two rings of circular section fireclay bricks (E). The lower or inlet end was a Carbolox plate (F) cemented into the end of the furnace and containing two openings. Into one was sealed the thermocople sheath (G) and into the other the silica inlet tube (H) for chlorine. The latter was fitted with a brass flange which connected with the chlorine supply. The upper or outlet end consisted of another Carbolox plate (I) leading to a side outlet tube (J) of Carbolox bearing a flange (K). This end plate was removable, and fitted tightly into the upper end of the furnace. After charging it was clamped down by means of the wing nuts (L) and luted round the joint with a cement made from sodium silicate and finely divided porcelain.*
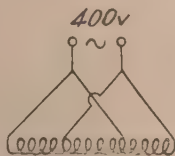
Fig. 6.—Wiring diagram of pilot plant furnace.

The whole furnace as so far described was mounted by means of trunnions on a rigid stand. In this way it could be tilted to any desired angle for emptying, and was operated in the vertical position.

The charge rested on a bed of coarse silica chips (N). The bed was about 8 in. in depth and served also as a preheating zone for the chlorine.

The chlorine was supplied from a number of 250-lb. cylinders connected into a manifold. From the manifold the gas passed through an orifice-type flowmeter graduated in pounds per hour, and thence to the inlet of the furnace (H) via a T-piece (O) of Pyrex glass. This T-piece was connected to a manometer on the control panel and indicated the internal pressure at the inlet end of the furnace. Also on the control panel was the ammeter in the electrical heating circuit, a large variable resistance (20 ohms-20 amps) for controlling the temperature of the furnace, the pyrometer indicating this temperature and the above-mentioned chlorine flowmeter.

*Mica and Insulating Supplies Co. Ltd. market a product called " Miscolute " which has been found very satisfactory. It must, however, be applied before the end of the furnace reaches 100°C.

The temperature of the furnace could be maintained at any desired value up to 1,000°C. Electrical heating was found to be even, clean, easy to control, and very convenient.

At the outlet end of the furnace the flange (K) was bolted to a 2-in. internal diameter lead pipe (P) which was connected to the condenser (Q). This consisted of a number of ¾-in. lead pipes (R) opening at the top into a lead-lined head (S) and at the bottom into a similar head (T) which carried the off-take pipe (U) for the waste gases and opened at the bottom into the collecting vessel for the tetrachloride (V). The lead tubes of the condenser were jacketed by a mild steel cylinder through which flowed the cooling liquid. The off-take (U) was connected to a glass bulb 10 in. in diameter, which served as a trap for titanium tetrachloride droplets, and also allowed the operator to see if excess chlorine was passing through the furnace. In fact, with experience, the appearance of this bulb conveniently indicated the state of operation of the furnace. On the outlet side of (W) was fitted a T-piece which was connected to a second manometer. This showed
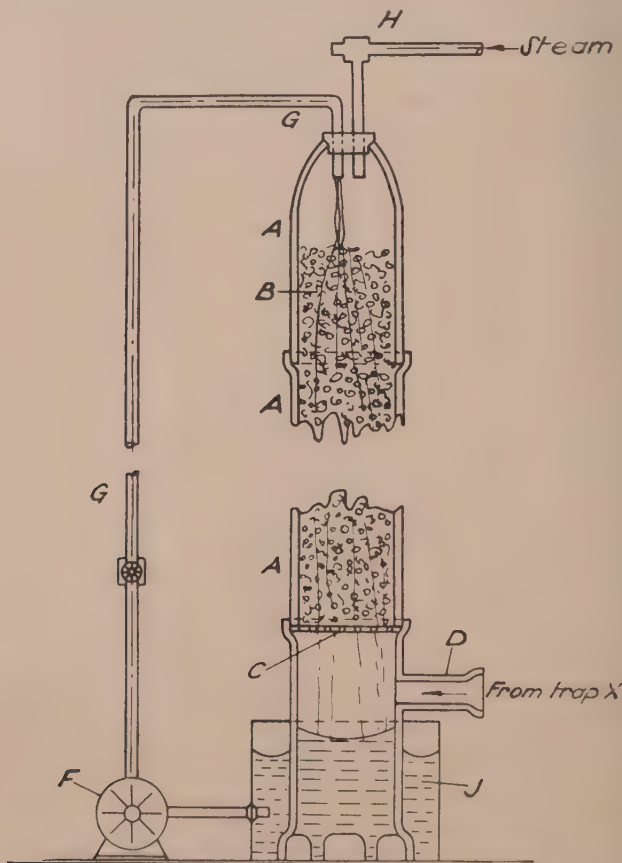
Fig. 7.—Diagram of absorption tower of pilot plant.

the internal pressure at the outlet end of the system. From (W) the
waste gases passed through a lead trap (X)—simply a sheet lead
box 8 in. x 8 in. x 12 in., which served to trap any water that splashed
back from the absorption tower. The latter, which is shown in Fig. 7,
consisted of four 12-in. internal diameter glazed earthenware pipes (A)
cemented together to form a tower approximately 12 ft. in height and
packed with porcelain rings (B), the rings being supported on a
perforated earthenware plate (C) situated just above the waste gas
inlet (D). The tower stood in a 100-gallon tank (J) which contained
a strong solution of sodium carbonate. This was circulated through
the tower by means of pump (F) and pipe (G) and served to absorb
any excess chlorine that might pass through the system, together with
uncondensed titanium tetrachloride vapour, traces of hydrochloric acid
gas, &c. Into the top of the tower were sealed the pipe (G) and a
steam ejector (H), and by means of the latter a slightly reduced pres-
sure was maintained throughout the whole furnace system. This
reduced pressure was indicated by the two manometers previously
described. Normally, both register the same pressure, but should a
blockage occur anywhere in the furnace, pipes, or condenser, a differ-
ence in pressure would occur. Thus the manometer on the inlet side
was a very sensitive "blockage" indicator. It also served to indicate
difference in pressure caused by an accumulation of dust in the furnace
towards the end of a chlorination.

(ii) *Experimental Procedure.*

For the pilot plant preparation of briquettes porous masses were
made in 80-lb. batches by spreading out the tarry paste resulting from
the mixing of 60 lb. rutile, 20 lb. coal, and 35 lb. tar to a thickness of
about $\frac{3}{4}$ in. in an iron tray, 5 ft. 6 in. x 2 ft. 6 in. with a 3-in. side, and
burning off the tar with the aid of a wood fire beneath the tray. On
cooling, the cake was broken into approximately 1-in. cubes which were
ignited to 700°C. in an atmosphere of nitrogen until all evolution of
vapour had ceased.

After coarse silica chips had been placed in position in the bottom of
the furnace the charge (about 40 lb.) was introduced, and the top plate
placed in position, clamped down and luted. Heating the charge from
room temperature to 700°C. took 3½ hours, the current being 16·5
amps. at 400 volts. If the charge was hot when added this time was
considerably reduced. Meanwhile, the condenser, collecting vessel,
outlet pipes, &c., had been connected, and immediately prior to passing
chlorine, the steam ejector was turned on until the manometers (which
should at this stage give the same reading) registered a reduction in
pressure in the furnace of about 2 in. (water pressure). The cooling
liquid, which was tap water, ice-cooled water at 3-10°C., or refrigerated
brine at -10°C., was passed through the condenser and the chlorine was
turned on at 5–7 lb. per hour. Titanium tetrachloride was evolved
almost immediately, and condensation began some 10 minutes later; 7–8
lb. was collected per hour. The temperature of the furnace was held at
700°C., but was not critical, as has already been shown, and could
vary between 600° and 800°C. without harmful effects. Whitish or
pink fumes could be observed passing through the glass bulb, and these
persisted throughout the run no matter how carefully the briquettes
and furnace were dried before chlorination. This suggests that there

is a certain amount of combined moisture in the calcined coal which is not freed by heat alone, but is only evolved when actual chemical breakdown of the material occurs. During the course of the chlorination, which usually took 7–9 hours, the chlorine rate was gradually reduced until, when 75–80 per cent. of the charge had been used, the rate was about 3 lb. per hour. Also towards the end of the experiment a difference in pressure was observed between the two manometers; that at the outlet remained at –2 in. throughout, while that at the inlet end finally read from –1 in. to –1½ in., depending on the amount of dust in the bottom of the furnace. A difference of 1 in. was the most that was ever observed, and if the charge was chlorinated to the extent of over 85 per cent. the pressure difference became smaller owing to the small amount of charge remaining in the furnace. Finally, the chlorine was turned off and nitrogen was blown through for half an hour to displace the remaining titanium tetrachloride vapour and clear the furnace, which could then be opened. The titanium tetrachloride collected at the beginning of the run was usually a pale straw colour and contained little or no suspended ferric chloride. Later the colour darkened owing to the formation and condensation of this compound.

Care was taken to ensure that there were no air leaks in the system. Should there be a leak at the inlet end of the furnace the air that would be drawn in would cause combustion of the carbon and low yields. If there were a leak at the outlet side, but before the condenser, the titanium tetrachloride vapour would be diluted and would tend to be swept through without being condensed. Leaks in the piping, &c., on the outlet side of the condenser would not be so serious, but it was desirable to eliminate *all* leaks. However, should there be one or more small leaks, the reduced pressure within the system would prevent escape of objectionable chlorine gas and fumes. Equally important was the drying of all piping, &c., before use and the careful checking of the condenser for water leaks. Even a very small quantity of water would react with titanium tetrachloride to form a voluminous yellow mass which could entirely clog the system.

TABLE 1.

Expt. No.	Rutile in Briquettes.	Charge Weight.	Weight of Charge not Used.	Percentage Used.	Cl_2 Used.	Cl_2 Efficiency.	$TiCl_4$ Expected.	$TiCl_4$ Condensed.	Condenser Efficiency.	Temperature of Cooling Water.
		lb.	lb.		lb.	%	lb.	lb.	%	°C.
8	Ground ..	44	5·5	87·5	57	90	68·2	67	98	−10
9	Ground ..	40	15	62·5	35·8	94	45	40	90	11
11	Unground	38	20	48	28*	90	33	30·5	91·5†	−10
12	Unground	30	15	50	21·5	93	27	26·2	97	10–12

* Chlorine was passed too rapidly at the beginning of the run.
† A little TiCl₄ was lost owing to water leaking into the collecting vessel.

(iii) *Chlorine Efficiency.*

The figures set out in Table 1 are typical of pilot plant operation.

The figures for the chlorine efficiency are based on the titanium dioxide (TiO_2) used, i.e., on the difference between the weight of TiO_2 in the initial charge and that found by analysis in the residue. Efficiencies thus calculated are the true ones from the stoichiometric chemical viewpoint. However, from the practical viewpoint it is the amount of tetrachloride condensed that determines the overall efficiency of the process. Hence, in separate columns are given the tetrachloride expected (based on the weight of oxide consumed) and that actually condensed. From these values the *condensation efficiency* is calculated. It will be noted that there is considerable difference between the condenser efficiencies obtained in experiments 8 and 9 owing to the use in the former of refrigerated brine at $-10°$C. in place of water at $11°$C. in the latter. However, the 10 per cent. loss in No. 9 and at other times when cooling water from $10°-20°$C. was used, appeared to be due to mechanical " carry over " of the tetrachloride in the form of a fine mist of droplets rather than failure to condense the vapour. An attempt was made to test this hypothesis by connecting to the outlet of the condenser two traps consisting of 20-litre glass flasks. These in effect provided a settling space for the droplets and the results (see Expt. No. 12) show a degree of efficiency approximating that of No. 8.

The chlorine efficiency fell when the chlorination was continued beyond about 70 per cent. consumption of the charge. Thus, in No. 8, $87·5$ per cent. of the charge was consumed and the overall chlorine efficiency was 90 per cent.; while for No. 9. using a similar charge, but consuming only 63 per cent., an efficiency of 94 per cent. was obtained. This is, of course, due to the great reduction in depth of charge that occurred as the chlorination progressed. Initially, in the case of No. 8, the depth was about 3 ft. 6 in., whereas at the end it was only 5 in. Such a shallow layer of material could not be expected to give a good chlorine efficiency even at the reduced gas flow rate. It is therefore recommended that not more than 70–75 per cent. of the charge be used at one time, the residual material being reused in later batches. However, it is emphasized that this figure applies only to our pilot plant and may differ for other furnace designs.

Providing that the chlorine is not passed too rapidly through the charge it has been shown that an efficiency of at least 94 per cent. can be obtained. For larger furnaces of considerable depth, a better figure than this could be expected.

For ground rutile briquettes the optimum chlorine rate appeared to be in the vicinity of 6–7 lb. per hour, gradually falling to about 3 lb. per hour when 70 per cent. of the charge had been consumed. For the unground rutile, a slightly lower rate seemed necessary, say 5–6 lb. per hour. It is emphasized that here again the figures apply only to our furnace and would certainly not apply to any other size or shape of furnace.

Fig. 8.—General view of pilot plant.

(iv) *Use of Unground Rutile.*

The results obtained in the laboratory, which have already been discussed, have been substantiated in the pilot plant. The unground naturally-occurring sand proved almost as good for the production of titanium tetrachloride as the ground material. The chlorine rate, as stated above, had to be reduced slightly, but this drawback, in practice, might be more than compensated by the fact that there would be no necessity for grinding. There is only one other way in which the unground rutile suffered in comparison with the ground. This was in the formation of the porous pieces. The briquettes formed from unground mineral are not so strong as the others, and crumble more easily. This is shown by the fact that the 20 lb. residue from No. 11 consisted of 12 lb. of unbroken lumps and 8 lb. of dust, while that from No. 9 contained 11 lb. of lumps and only 4 lb. of dust. This reduction in strength is probably due to the relatively larger size of the grains and their smooth surface, there being no rough edges for the binder to grip. These briquettes have, however, proved quite satisfactory in the pilot plant furnace, and should they prove too crumbly in full-scale production the trouble could probably be overcome by mixing in a small **proportion of ground rutile.**

(v) *Purification of Titanium Tetrachloride.*

As stated previously, the tetrachloride obtained from the pilot plant furnace is a deep red colour, due to suspended ferric chloride. At the commencement of each run, several pounds were condensed which were of a light straw colour, but this colour soon gave place to the deep red which became more noticeable as chlorination proceeded. If, however, the whole of the tetrachloride produced during the run was collected into a single large flask or other transparent vessel it was noted that ferric chloride settled after a few days to a thin layer on the bottom. The bulk of the tetrachloride was left crystal clear and bright yellow in colour, and could easily be siphoned off. In this way a large proportion could be obtained free from ferric chloride or other sediment. For example, in one such separation 14 litres of dark-red tetrachloride was allowed to stand for four days. At the end of that time 13 litres of crystal clear liquid was taken off (93 per cent.). The remaining litre was then distilled and yielded a further 800 cc. of clear $TiCl_4$ and 375 grams of a dark-red sludge consisting of ferric chloride and titanium tetrachloride. This unusable residue amounted to only 1·5 per cent. of the whole.

For analysis three samples were prepared from the bulk tetrachloride obtained from a typical chlorination. These were:—

1. Clear yellow liquid obtained as described above by decantation from the crude furnace product.

2. The clear yellow liquid of sample 1 was subjected to a single distillation. The distillate was an orange liquid of a deeper tint than the original.

3. The deep coloured liquid of sample 2 was refluxed over copper foil until clear and was then distilled from the copper. The distillate was crystal clear and water white.

Table 2 gives the results of the analyses in summarized form, together with the specification for titanium tetrachloride.

TABLE 2.—ANALYSIS OF TITANIUM TETRACHLORIDE FROM PILOT PLANT.

Analysis for	Sample 1.	Sample 2.	Sample 3.	Specification Limits.
Description.	Crystal bright yellow.	Crystal orange-yellow.	Crystal water white.	Clear liquid ; colorless, yellow or red.
Specific gravity at 15·5° C.	1·727	1·732	1·732	1·72–1·74
Ti as TiCl₄	99·1	99·4	100·2	Not less than 95 per cent
Si as SiCl₄	0·51	0·30	0·17	Not more than 5 per cent
Total chloride	74·6	74·6	75·0	74·6–75·5 per cent.
Fe as metallic Fe	< ·02	< ·02	Not detected	Not more than ·04 per cent.
Excess Cl₂ as chlorides	⟫0·25	⟫0·18	..	Not more than 0·5 per cent.
Free chlorine	0·15	0·12	<0·01	Not more than 0·2 per cent.
Residue on evaporation	Non-detectable			Not more than 0·25 per cent.

5. Analytical Methods.

(i) *Manipulation.* A method was developed which overcame the difficulty of handling the mobile and intensely fuming titanium tetrachloride, and in particular the difficulty of weighing samples for analysis. A thin walled ampoule was blown from thin glass tubing (3/16 in. external diameter) and sealed off under vacuum, care being taken to leave a neck about 1 in. long on the bulb and a long capillary at the point of sealing. The ampoule was then weighed and the sealed stem was next immersed in the sample and the tip broken with a small pair of tweezers. When the bulb was filled it was removed from the tetrachloride, cleaned by a stream of dry air which evaporated the tetrachloride remaining on the stem, resealed in a micro flame and again weighed. A number of checks were made on the amount of glass lost when the tips of the bulbs were broken. In no case was this found to exceed 2 mg., and the average was below 1 mg. Samples used weighed approximately 1 gram, so that the error introduced by the loss of glass can be made quite small. For samples weighing 8–10 grams it was negligible. These bulbs were broken, as desired, under solutions of NaOH or 4N HCl in sealed flasks.

(ii) *Total chlorides* were determined by taking up the sample of tetrachloride in NaOH of approximately 0·5 normality, boiling, filtering off the precipitate of Ti (OH)₄ and titrating the filtrate with AgNO₃ using chromate indicator.

(iii) *Free Chlorine.* 8–10 gram samples were used, and were taken up in 4N HCl in glass-stoppered bottles. After all fume and cloudiness of solution had disappeared about 0·5 g. KI was added and the iodine liberated was titrated with N/100 thiosulphate using starch indicator.

(iv) *Silicon Tetrachloride*. The silicon was estimated colorimetrically using ammonium molybdate solution. This standard method for silicon had to be slightly modified for our purposes. At a pH of 1·2–2 (at which the yellow complex molybdate colour has to be developed) titanium hydroxide commences to precipitate. This would, of course, prevent comparisons of the colour being made. However, it was found that *after the colour had developed* the solution may be made more acid without affecting the colour in any way. In this way, by the addition of a few drops of concentrated H_2SO_4, the $Ti(OH)_4$ could be dissolved leaving the clear yellow colour. The presence of the $Ti(OH)_4$ while the yellow colour developed did not have any adverse effect.

(v) *Iron*. Iron was estimated colorimetrically by means of thiocyanate. A point of interest regarding the coloration of the crude titanium tetrachloride was noted here. It appears that less than 0·02 per cent. of iron as ferric chloride causes development of a bright yellow colour and regarded as a test for ferric chloride this method is more sensitive than the thiocyanate method. Furthermore, the saturation concentration of iron (as $FeCl_3$) in $TiCl_4$ has been found to be less than ·02 per cent. at 20°C., so that providing there is no suspended material present the amount will always be within specification limits.

(vi) *Titanium*. Titanium was estimated by the cupferron precipitation. As the iron was negligibly small in all cases there was little need to separate the titanium from the iron in the standard manner.

Inspection of Table 2 shows that all the samples of the tetrachloride conformed to specification—even the crude furnace product, provided the $FeCl_3$ was allowed to settle out. This is a gratifying feature, as it means that a very minimum of purification is necessary, the process of settling and decantation presenting no difficulties for full-scale production.

6. Cost of Raw Materials and Electrical Power.

The figure given is based on the following costs and assumptions:—

Chlorine in ton quantities (or more)	£35 per ton
Black bituminous coal	£2 3s. per ton
Cost of grinding coal to – 200 mesh	£2 per ton

The chlorine efficiency is 90 per cent.

The condenser efficiency is 95 per cent.

The cost of unground rutile is	£35 per ton

   The rutile contains 95 per cent. TiO_2

Tar ..	£8 per ton
Electrical power	1d. per kilowatt hour

$$Tar + TiO_2 + 2C + 2Cl_2 = TiCl_4 + 2CO—$$

0·6 ton + 1 ton + 0·3 ton + 1·8 ton = 2·38 ton   Theoretically

0·6 ton + 1·05 ton + 0·3 ton + 2·0 ton = 2·18 ton   Corrected for efficiencies

£4 16s. + £36 15s. + £1 5s. + £70   = £112 16s.

Heat input provided by electrical heating element:

    (*a*) Heating from room temperature to 700°C. 3½ hours at 16·5 amperes and 400 volts = 23·1 kilowatt hours.

    (*b*) Maintaining temperature during the reaction—a total of 4 hours "current on" operation = 27·5 kilowatt hours.

Total power consumed—50·6 kilowatt hours, during which approximately 60 lb. of titanium tetrachloride would be produced = approximately 1d. per pound.

Total cost for materials and power is therefore approximately 7d. per lb.

It must be emphasized that these figures are only approximate, as costs are fluctuating and quotations that we have received from time to time varied considerably. This applies particularly to rutile.

7. Conclusion.

It has been shown that naturally occurring Australian rutile sand could well be used in place of titanium pigment for the production of titanium tetrachloride. This would result in a considerable reduction in the number of operations making up the process together with a reduction in cost.

Details of the process have been given, including preparation of the raw materials, materials of construction of the furnace, condenser and other parts of the plant; data concerning the efficiency of chlorination, condensation, and in general, the chemical and physical aspects of the reaction.

8. Acknowledgments.

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A Survey of Houses Affected in the Beaumaris Fire, January 14, 1944.

By G. J. Barrow.*

Summary.

To determine the influence of the type and details of construction on the resistance of houses to external fire hazards, a survey was made of the damage caused by the fires which swept the bayside resort of Beaumaris, Victoria, on January 14, 1944. General observations on the 66 houses destroyed or damaged and a detailed study of seventeen representative houses showed that the resistance to fire is determined more by the details of construction than by the materials used in the walls. Although the damage was caused primarily by the external fire, practically all the houses ignited inside, i.e. in the roof space, in rooms, or under the floors, due to the ingress of flame, sparks, and embers through openings such as ventilators, eaves, and windows. Sealing or screening such openings by fine wire mesh greatly reduced the risk of damage, and the conclusion was drawn that, from the point of view of resistance to external fires, a house should be as air-tight as practicable and that any openings which cannot be eliminated should be screened. A list of recommendations, the adoption of which should do much to reduce the fire risk, is included.

1. Introduction.

On January 14, 1944, one of the worst conflagrations in the history of the metropolitan area of Melbourne, swept through the bayside resort of Beaumaris. Beaumaris is situated on the eastern coast of Port Phillip Bay, 12 miles from Melbourne; some twenty years ago it featured in a closer settlement scheme and an electric tramway service was extended from Black Rock. The scheme was abandoned and the tramway system, after operating for a period of five years from 1926 to 1931, was discontinued and subsequently dismantled. Since that date there have been no improvements made in the area, which, although subdivided in detail has been allowed to return to its natural state. The dwellings in the area are of two main types—

(i) Small four- or five-room houses of varying construction, built as seaside dwellings either at the time of the settlement scheme or since; because of the housing shortage most of them are now permanently occupied.

(ii) Large homes constructed either by the early settlers or, in recent years, by residents who are not dependent on public transport.

The fire destroyed 58 houses and damaged eight others, and advantage was taken of the opportunity to obtain general data on the fire hazard in semi-rural areas and to determine the influence of the type and details of construction on the resistance of houses to external fire hazards.

Approximately 100 houses were inspected during the week following the fire, and from these seventeen were selected as being representative of the general damage and conditions. A detailed study of these particular houses was then made.

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The number of persons interviewed who were actively involved in the fire (either owners, occupiers, or service personnel) was approximately 200, and from these eye-witnesses data were collected on matters relating to houses destroyed, damaged, or saved.

The gathering of the data for this report was greatly facilitated by the willing co-operation of the following:—Police officers at Mentone, Black Rock, and Cheltenham; the Town Clerk of the Sandringham City Council; the superintendent, staff, and linesmen of the Cheltenham District Depot of the State Electricity Commission; the district officer of the Melbourne and Metropolitan Fire Brigade; auxiliary and volunteer fire fighters; owners, occupiers, and agents of the damaged properties.

2. Location of the Fire.

The fire occurred within an area of some 1,500 acres, bounded on the north by Bay-road, Cheltenham, the west by Reserve-road, the south by the sea, and on the east by Charman-road, Mentone and Cheltenham, as shown in Map 1. The area north of Weatherall-road is included in Map 1 because, although there is little damage in this area, it is believed that the main fires through the Beaumaris area originated there. Of the 1,500 acres included in the map some 700 were burnt.

The main fire area south of Balcombe-road was mainly dense scrub land covered by a heavy growth of tea-tree (*Leptospermum laevigatum* and *L. coriaceum* mixed) with scattered groups of manna gum (*Eucalyptus viminalis*) and coast acacia (*Acacia sophorae*), and with groups of white sallow acacia (*Acacia floribunda*) further back from the coast; throughout this area were scattered clearings of from 1 to 5 acres densely covered with dry grass and bracken (*Pteridium aquilinum*) often to a height of 3 feet; in the lower swamp areas were several acres of swamp tea-tree (*Melaleuca squarrosa*). The houses destroyed and damaged extended throughout this area, but were mainly situated within three-quarters of a mile from the sea.

In attempting to retain the locality in its natural state, the majority of the residents had allowed their properties to become thickly wooded with native trees and shrubs, augmenting the natural growth in many cases with exotic vegetation, thus forming a dense thicket to within 10 to 20 feet of their dwellings, and in many cases in contact with them.

Scrub fires in this area have been a common occurrence for many years, but are usually localized and are fairly easily checked before constituting a menace to the populated sections of the district. The origin and progress of the fire, as observed by officers of the State Electricity Commission, are shown in Map 1. It will be noted that there were two distinct fires: The first swept over the eastern portion of the burnt area, and the second, which occurred half an hour to an hour later, burnt the western portion, overlapping to some extent the area covered by the first fire. Both fires burnt down to the water's edge.

As will be seen from Map 2, the fire burnt over both scrub land and grass land, but was very much fiercer and presented a much greater hazard to houses in the scrub land. In the grassed areas, it was comparatively easily controlled by breaks such as roads and cultivated areas, and by spraying with garden hoses.

The fires were fought by personnel of the Metropolitan Fire **Brigade,** State Electricity Commission, Police Department, Civil **Defence** Organizations, Army, and local residents and volunteers with fire hoses, garden hoses, and, where the water supply failed, with beaters. As a result of their efforts, a number of buildings which were apparently doomed were either saved or only slightly damaged.

About 208 houses in the area were in some danger, but 90 of these, located in cleared areas or in grass lands, even though in some cases completely surrounded by fire, were not in immediate danger, as the fire was controllable and was deflected away from the houses by the fire fighters. The remaining 118 houses were gravely threatened because of actual contact with flames or burning debris.

3. Conditions Leading up to the Fire.

Meteorological data for the day of the fire, together with the weather conditions for the preceding six months, as supplied by the Commonwealth Meteorological Services, Department of Air, Melbourne, are given in the Appendix.

Conditions prior to and during the fire were ideal for a major conflagration. The six months preceding the fire were abnormally dry and several perennial species of small flora (particularly sun-dews and orchids), for which this and surrounding districts are noted, had failed to appear. The day of the fire was hot with a strong dry northerly wind, the maximum shade temperature being $103 \cdot 2°$ F., and the maximum wind velocity being 54 m.p.h., the relative humidity falling to 6 per cent., corresponding to an equilibrium moisture content in wood of about 2 per cent.

4. Extent of Damage and Location of Buildings.

The location of the houses in the affected area, together with information on the type of construction and the extent of the damage (if any), is given in Map 2.

For convenience in locating the seventeen houses selected for detailed study, Map 2 has been divided into sections A, B, and C. Sections A and B have each been subdivided into three sub-sections.

The extent of the damage in relation to the type of construction and number of houses seriously threatened by the fire is given in Table 1. It may appear from this table that the brick and concrete houses were less liable to danger than timber frame houses with fibro-cement or ash-cement rendered walls. However, statistical investigation has shown that there are no significant differences in the percentages of different types of houses destroyed or damaged.

It must also be noted that the brick, brick veneer, and concrete houses were in general located somewhat more favourably than the others, in that they were generally surrounded by well-kept gardens, which tended to lessen the severity of attack. Brick houses which were

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completely gutted, leaving the walls only standing, have been considered as being destroyed in compiling the Table (see Plate 1, Fig. 1). Further details of the extent of the damage in relation to the type of construction and location of the houses are given in Table 2.

TABLE 1.—NUMBER OF AFFECTED HOUSES IN RELATION TO TYPE OF CONSTRUCTION AND NUMBER SERIOUSLY THREATENED.

Construction.	Number Seriously Threatened.	Number Affected.			Percentage Affected.		
		Destroyed.	Damaged.	Total.	Destroyed.	Damaged.	Total.
W : $\frac{W}{B}$ ..	83	41	5	46	50	5	55*
$\frac{CS}{W}$: CS : $\frac{ACL}{W}$	15	8	2	10	53	13	66*
B : V : C ..	20	9	1	10	45	5	50*
Total ..	118	58	8	66	..	..	..

NOTES.—1. An additional 57 properties sustained damage to outhouses and fences only

2. Statistical examination has shown that there is no significant difference between the percentages marked thus *.

LEGEND.—W—weatherboard.

$\frac{W}{B}$—weatherboard with brick foundation.

$\frac{CS}{W}$—timber frame with weatherboards to 3-4 feet from ground and fibro-cement sheets above.

CS—fibro-cement sheet over timber framework.

$\frac{ACL}{W}$—timber frame with weatherboards to 3-4 feet from ground and ash and cement rendering and lath construction above.

B—brick and brick stucco covered.

V—brick veneer.

C—concrete block or slab.

5. Observations.

Points of particular interest are illustrated by the photographs which, with the accompanying captions, are self-explanatory.

Detailed records of the construction, surroundings, point of ignition and extent of the damage to the seventeen representative houses mentioned previously have been filed and are available to any one interested, but the following observations should be of general interest.

The use of ½-in. mesh wire netting as a guard against birds, rodents, and possums proved to be of great value in preventing burning debris from being carried by draughts through wall ventilators, louvre openings, and under floor air-vents (see Plate 5, Fig. 1).

The fire burnt the fences of fourteen houses but was stopped before it could constitute a menace to the houses, which were situated either in cleared areas or in grassland.

TABLE 2.—HOUSE CONSTRUCTION AND EXTENT OF DAMAGE IN EACH SECTION.

Section.	Construction.		Destroyed.	Damaged.	Out-houses, Fences Damaged.	Undamaged.
A1 ..	Scrub and fence damage only		..	..	..	..
A2 ..	W ..	Corrugated iron roof ..	6	1	8	1
	W ..	Tile roof	2	..	3	..
	CS ..	Corrugated iron roof ..	2	..	1	..
	W̄					
	CS ..	Fibro-cement roof ..	1	..	1	..
	B ..	Corrugated iron roof ..	..	..	..	1
A3 ..	W ..	Corrugated iron roof ..	12	..	6	7
	W ..	Corrugated iron roof ..	1	..	..	..
	B̄					
	W ..	Tile roof	4	1	..	4
	CS ..	Corrugated iron roof ..	..	..	..	3
	W̄					
	B : V	Tile roof	2	1	1	2
	C ..	Corrugated iron roof ..	1	..	1	..
B1 ..	W ..	Corrugated iron roof ..	2	1	1	1
	W ..	Tile roof	..	..	..	1
	ACL/W	Corrugated iron roof..	..	..	..	1
B2 ..	W ..	Corrugated iron roof ..	4	..	8	5
	W ..	Tile roof	1	..	3	3
	CS/W	Corrugated iron roof..	..	..	1	3
	ACL/W	Corrugated iron roof ..	1	..	..	1
	B ..	Corrugated iron roof ..	..	..	..	1
	B ..	Tile roof	..	..	2	5
B3 ..	W ..	Corrugated iron roof ..	3	1	8	3
	W ..	Zinc-anneal roof ..	..	..	..	1
	W ..	Tile roof	3	..	3	4
	WCS	Tile roof	1	..	1	..
	ACL/W	Tile roof	1	..	..	..
	B ..	Zinc-anneal roof ..	..	..	1	..
	B ..	Tile roof	5	..	4	7
	V ..	Tile roof	..	..	4	..
C1 ..	W ..	Corrugated iron roof ..	2	1	5	20
	W ..	Tile roof	1	..	4	14
	W/B	Tile roof	1	..	..	..
	CS/W	Corrugated iron roof ..	1	1	..	4
	CS ..	Fibro-cement roof ..	1	..	1	1
	CS ..	Corrugated iron roof ..	..	1	..	2
	CS ..	Tile roof	..	..	1	..
	B ..	Tile roof	..	..	2	9
	B ..	Corrugated iron roof ..	..	..	1	7

Fly-wire proved in a number of cases to be an excellent spark arrester. House B3-6 being a case in point. This dwelling had large

openings across the front covered by fly-wire; immediately behind the wire were large canvas blinds which were down during the whole period of the surrounding fire. A general profusion of sparks and burning material was swept against this portion of the house, but an examination of the framing ledge between the fly-wire and the blinds showed it to be free of any charred material or ash.

Both hessian and canvas showed little resistance to sparks, hessian bags lying in yards and canvas blinds up to a distance of a mile from the fires being burnt.

Wall ventilators and air vents below floor level, where unprotected by some form of screening, proved disastrous in a number of cases. In some cases where the fire appeared to be controlled a sudden draught swept sparks up to the dwelling and in through these vents. This point was well illustrated in the fighting of the fire at House B2-1. While a fire was being fought in the eaves and ceiling at the rear of the house, the draught through the underfloor vents near the ground at the back of the house swept burning material under the house to such an extent that a fire under the floor became uncontrollable. A similar type of attack occurred at Houses B3-9, C1-1, and C1-3.

Houses with eaves either boxed with fibro-cement sheets or completely boarded had a greater degree of safety than those with eaves left open for ventilation. It was reported that the majority of fires started in the roofs.

Fibro-cement sheets were found to be fire retardant but not fire proof; when subjected to intense heat they cracked, flaked, and collapsed. Where water could be poured or sprayed to keep them cool they proved a most effective check to the spread of fire. This point is shown in Plate 2, Fig. 2, the fibro-cement sheets are still intact above the steel door of the garage. The fire within the garage was so fierce that the front of the garage was sprayed with a fire hose for more than fifteen minutes before the fire behind the door burnt out.

Several fires became uncontrollable because shingles on the gable ends of the buildings ignited and the draught swept the flames into the roof through large roof ventilators. It would be preferable to replace these by a number of small ventilators, as shown in Plate 6, Fig. 1.

Badly fitting Marseilles pattern tile roofs were a source of danger; houses which either had a reasonably good chance of escaping the fire or were considered to be saved as the fire had passed, were either damaged or destroyed through the access of sparks and small burning debris under the tiles.

There were several cases of roof fires in houses roofed with corrugated iron; generally these fires entered through large roof ventilators in gable ends or ignited the protruding eaves and burnt through to the rafters and ceiling joists. The position of the guttering prevented the updraught from carrying the sparks up under the corrugations into the roof, however it is a possibility that in some cases an accumulation of burning debris, lodged in the guttering, may have been swept under the corrugations by an eddy of wind.

Compared with that of sealing a tile roof with its multiple openings, there is little difficulty in sealing a corrugated iron or corrugated fibro-cement roof. This can be done by scalloping the fascia board to fit the corrugations, by nailing a scalloped sheet of iron to the fascia, or filling the spaces under the corrugations with fibro-cement or mortar.

Throughout the fire several houses were seen to ignite although there was no apparent contact with the flame, sparks, or burning debris; this ignition took place both in the roofs and in the walls.

In a number of cases the proximity of high trees, preceded by very low scrub or grass, proved an effective fire break. This applied only in cases where branches of the trees were not located under the eaves. Due to the hurdling effect of the high trees the draught caused by the fires swept the burning material over the house. In the majority of cases the trees either failed to ignite or burnt slowly and so proved controllable (Fig. 1 and Plate 6, Fig. 2). It must be emphasized that high trees gave protection only when surrounded by grasslands or by low scattered scrub.
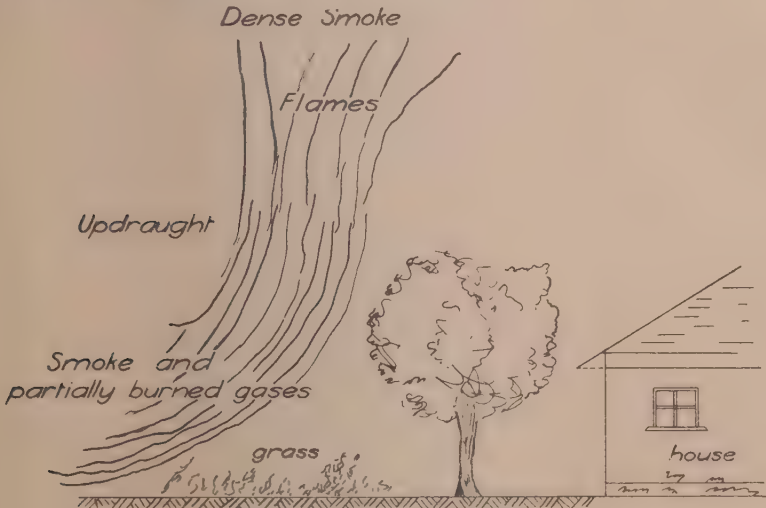
FIG. 1.

In the case of unattended houses (where fire fighting was impossible), the normal time from ignition to destruction was seven to fifteen minutes for brick or brick veneer and ten to 25 minutes for timber frame houses.

Evidence obtained during the survey indicates that there is no possibility of predicting the behaviour of a scrub fire. In a number of cases, scrub and trees which appeared to be burnt out, re-ignited, accompanied by a crackling and hissing noise suggestive of gas igniting; these flashes of fire would then jump distances up to several hundred feet igniting any scrub through which they passed. The rush of flame and the intense heat of the burning tea-tree caused many unexpected draughts and caused runs of fire to burn back against the wind for several hundred yards.

This process of the fire chopping about in several directions was instrumental in saving some houses by first burning breaks either near or partially around them. This occurred in the case of House B3-1 where a fire burnt off the scrub to the front, the east, and a section of the trees to the south (rear) of the house, thus leaving only one side and a corner to be protected when the second fire came through from the north-west. Inspection of this dwelling after the fire gives the impression that it was completely surrounded by fire. This was quite correct, but the fire was not burning on all sides at the same time.

6. Conclusions.

The survey showed that, in a fire of the type that swept Beaumaris, the chances of a house surviving are determined more by the nature of the surroundings and the details of construction than by the materials used in the walls. With two exceptions, all the really destructive fires started inside the houses, i.e., in the roof space, in rooms, or under the floors, the immediate cause of ignition in such cases being the entrance of flame, sparks, and burning debris through openings such as ventilators, eaves, and windows. An air-tight house would appear to have much greater resistance to fire than one in which free circulation of air is encouraged. Of course, an air-tight house, even if it could be built, would be most undesirable for other reasons, but it is necessary in areas such as Beaumaris, where a serious fire hazard exists, to make a compromise between fire resistance and ventilation, by completely boxing the eaves, sealing roofs, &c.

In the two exceptions referred to previously, the houses appeared to ignite externally due to the high temperature of the surrounding scrub fires. However, these observations may not be correct, as under conditions such as existed at the time, observer's reports are often unreliable, and it is quite possible that these houses also caught alight because of the entrance of flame and sparks.

Experience at Beaumaris showed that the ill effects of openings can be considerably mitigated by covering them with $\frac{1}{2}$-in. mesh wire netting or preferably with fly-wire. The wire netting will prevent the entrance of burning debris, and fly-wire is effective in stopping sparks. These coverings, in a number of cases, saved houses from destruction. Similarly, fly-wire window-screens and doors are of great assistance in preventing the entrance of sparks in the event of windows or doors being accidentally left open during the fire.

Badly fitting Marseilles pattern tile roofs appear to be a menace as they provide innumerable openings through which sparks may enter. Some form of sheet roofing, e.g. corrugated iron, fibro-cement, or close fitting composition roofing, appears to be much more satisfactory. Corrugated roofing should be sealed at the eaves, ridge, hips, and valleys by some such composition as mortar or fibro-cement.

These simple precautions greatly increase the resistance of a house to fire, with little, if any, increase in cost, and they are likely to be far more effective than such methods of protection as the use of non-combustible materials for walls. The impression gained from a glance at a completely gutted brick house in which the walls are still standing (see Plate 1, Figs. 1 and 2), is that the damage is much less serious

than in the case of a timber frame house which is usually burnt to the ground. However, a little consideration will show that the cost of the walls of a timber frame house is a comparatively small proportion of the total cost, and the cost of reconditioning a burnt-out brick house is likely to exceed the cost of rebuilding a timber frame house.

7. Recommendations.

The adoption of the following recommendations in areas where there is a fire hazard, would do much to reduce the risk:—

1. In timber houses, the walls below floor level should be close boarded, ventilation being provided by woven wire vents.

2. All vents should be of the woven wire type or else covered by a fine mesh.

3. Large ventilators in gable ends should be eliminated and replaced by a number of scattered small ventilators with fine mesh openings.

4. Eaves should preferably be completely boxed, but if left open should be covered by fine mesh wire netting.

5. Badly fitting Marseilles pattern tiles are a source of danger.

6. The space under the corrugations of corrugated roofing should be closed at the eaves, ridges, hips, and valleys.

7. Fly-wire window-screens and doors are beneficial.

8. Trees and shrubs should be kept clear of the walls.

9. Stacks of fuel should be well clear of the walls or stored in properly•constructed sheds.

Appendix.

WEATHER CONDITIONS IN MELBOURNE.

(Compiled from data supplied by the Commonwealth Meteorological Services, Department of Air, Melbourne.)

(1) CONDITIONS FOR SIX MONTHS ENDING JANUARY 31, 1944.

Month.			Mean Temperature.		Rainfall Points (100 points = 1″).	
			Temperature.	Normal.	Rainfall.	Normal.
			Deg. F.	Deg. F.		
August	..	..	47·9	51·0	95	189
September	..	..	53·1	54·2	199	228
October	..	..	56·8	57·8	84	262
November	..	..	60·3	61·4	312	227
December	..	...	63·7	64·9	68	229
January	..	..	68·6	67·4	74	193
Total	..	..	..	..	832	1,328

APPENDIX—*continued.*

(2) CONDITIONS FROM JANUARY 1 TO 14, 1944.

Date, 1944.			Maximum Temperature.	Maximum Wind Velocity.	Wind Direction (Prevailing).	Rainfall Points (100 Points = 1″).
			Deg. F.	Miles/hr.		
January	1	.. ..	100·6	40	NNE	nil
	2	.. ..	79·8	37	WSW	5
	3	.. ..	74·5	37	NNW	nil
	4	.. ..	68·7	42	SSW	2
	5	.. ..	69·2	37	WSW	16
	6	.. ..	72·9	23	SSW	1
	7	.. ..	84·0	26	SSE	nil
	8	.. ..	102·0	41	NNE	,,
	9	.. ..	74·4	29	S	,,
	10	.. ..	80·5	40	NSW	,,
	11	.. ..	69·9	26	W	,,
	12	.. ..	69·8	29	ESE	,,
	13	.. ..	91·5	18	WNW	,,
	14	.. ..	103·2	54	N	,,

(3) CONDITIONS ON JANUARY 14, 1944.

Times.			Condition.				
Local Mean.		Australian Eastern Daylight Saving.	Wind Velocity (Average).	Wind Direction.	Temperature.	Relative Humidity.	Equilibrium Moisture Content.
			Miles/hr.		Deg. F.	%	%
7	.. ..	8·20	22	N	80·4	25	5
8	.. ..	9·20	22	NNE	85·1	24	5
9	.. ..	10·20	26	N	88·2	17	3½
10	.. ..	11·20	28	N	91·1	13	3
11	.. ..	12·20	26	N	93·6	12	3
12	.. ..	1·20	24	N	97·1	10	2½
13	.. ..	2·20	22	N	99·7	7	2½
14	.. ..	3·20	23	NNW	101·2	9	2½
15	.. ..	4·20	20	N	103·2	9	2½
16	.. ..	5·20	16	N	101·1	8	2½
17	.. ..	6·20	16	NNW	101·1	6	2

(*a*) Maximum temperature at Melbourne (12 miles NNW of Beaumaris) on January 14, 1944, was 103·2° F. occurring at 14·55 L.M.T.

(*b*) The maximum gust of wind at Melbourne on January 14, 1944, 54 m.p.h. occurring at 10·07 L.M.T.

(*c*) The following is a description of the weather at Melbourne on January 14, 1944 :—

"Clear at first, overcast with cirrus cloud at 07·40 L.M.T. Almost cloudless at 1040. Dust and smoke haze greatly reduced visibility. At 1640 L.M.T. dense dust haze all around. At 1940 L.M.T. there was a dense dust haze. Visibility very poor."

Unavailability of Plant Food and Take-all of Wheat.

By H. R. Angell, Ph.D.[*]

Summary.

1. In an experiment that was made in five-gallon drums of soil out of doors during the seasons 1941-43, significant differences in the amount of damage by take-all were associated with differences in available plant food.

2. In the second and third seasons a significantly higher percentage of white heads occurred in the drums of soil to which neither chemicals nor inoculum were added in the first year than in the corresponding inoculated drums.

3. The association of white heads and take-all damage in drums with the isolation of *Ophiobolus graminis* from plants before jointing was significant.

4. Under the conditions of this experiment, the addition of nitrate nitrogen to the soil had little, if any, influence on take-all.

5. Small quantities of lime were without effect on take-all, large quantities reduced the effect of the disease presumably by making plant food available.

1. Introduction.

The influence of faulty nutrition on fungal infection of plants has engaged the attention of several workers whose contributions have been reviewed by Garrett (1939) and by Chapman and Brown (1942). Garrett (1942) has also quite recently reviewed the literature on take-all of wheat; consequently, at present, it appears unnecessary to attempt to relate the results here reported to the work of other investigators of this widely distributed and economically important disease of wheat.

The absence of take-all from drums in which the availability of plant food was increased by the addition of rather large amounts of lime was reported in an earlier paper (Angell, 1943). In contrast, marked reduction in yield was caused by the disease in drums containing

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only the native soil from which most of the available plant food was robbed by repeated cropping. In the third and fourth years of the experiment, the significant differences between the extent of crop reduction in the control drums and in those to which inoculum was added in the first and second years, appeared from the data to be due mainly, if not altogether, to differences in the amounts of available plant food left in the previous years by the larger crops in the control drums, and the smaller crops in the contaminated drums respectively. From the results of the experiment reported in this paper, it appears that some other factor or factors may contribute to this result.

Under the conditions prevailing in the wheat-growing areas of Australia, the more obvious symptoms of take-all usually appear from the time the ear is about to emerge from the boot to just before maturation of the grain. During this period death of what were apparently healthy plants followed by change in colour from healthy green to ashy grey, may sometimes take place within two days. Insidious and perhaps more economically important phases of the disease occur in many plants in the neighbourhood of the dead ones. Although such plants may appear healthy, they are in reality struggling on only a few more or less diseased roots to reach maturity. The grain borne by them is usually much lighter and smaller than the plump grain from healthy plants.

As far as the writer is aware, repeated reproduction in pot experiments of the above-described symptoms of the disease as they occur in the field has not been reported by other writers. The usual criterion employed by pathologists in evaluating damage by *O. graminis* and other organisms that are associated with take-all has been the extent of root rotting in the seedling stage. That this criterion is unsatisfactory is evident from the fact that rotting of the roots of seedlings that is induced by artificial inoculation has not necessarily been followed by comparable reduction of the yield of grain, or by other symptoms. Furthermore, under field conditions the extent of take-all damage at maturity can rarely be predicted by the appearance of the plants in the pre-jointing stage, unless soil conditions have been particularly unfavourable. In the experiments here reported, all the usual field symptoms have been obtained by providing some of the conditions that obtain in fields that are cropped to wheat year after year. The results indicate that conservation of soil fertility should be the underlying principle of the methods employed in the control of take-all.

2. Materials and Methods.

(i) *In* 1941.

Twenty tons of soil were gathered from an alluvial deposit on the Molonglo River in Canberra during February, 1941. The soil was sieved, quartered, thoroughly mixed, and then transferred to drums, each holding about 37·5 kilos. Three lots of eighteen drums were set aside as controls.

The remaining drums were also divided into groups of eighteen. Sulphur or the carbonates or sulphates of calcium, magnesium, potassium, or sodium were then added to each drum of a particular group as follows—

(a) Insoluble or relatively insoluble substances:

Sulphur	2 g.
Burnt lime, partly carbonated	250 g.
Burnt lime, partly carbonated	15 g.
Ground limestone	250 g.
Ground limestone	15 g.
Calcium sulphate	250 g.
Calcium sulphate	21·8 g.
Ground magnesite	250 g.
Ground magnesite	21 g.

(b) Soluble compounds:

Magnesium sulphate	61·5 g.
Potassium sulphate	13 g.
Sodium sulphate	42 g.
Potassium carbonate	12·6 g.
Sodium carbonate	37·0 g.

The allotted amount of each insoluble substance was thoroughly mixed with all the soil from each individual drum, and each soluble compound with only the lowest quarter of soil. The soil was then replaced and firmed sufficiently to ensure that during the period of growth of the plants the level would not recede to more than about one inch below the edge of the container. (In very few instances did the soil settle more than was desirable.) When all the drums were filled, they were randomized in position in the enclosure.

During the 15th and 16th May, 1941, twelve grains of Nabawa wheat were sown in the three lots of eighteen drums containing only the native soil. To all the drums in one of the three series about 20 small pieces of viable inoculum of O. graminis were also added and to the other two series an equal quantity of sterilized inoculum. All the drums to which elemental sulphur or compounds of calcium, magnesium, potassium, or sodium were added were also sown with Nabawa wheat, viable inoculum of O. graminis being added to nine drums of each series and sterilized inoculum to the remaining nine drums of each series. At the time of sowing the seed one gram of superphosphate was added to every drum in the enclosure.

Before shooting began, all but six seedlings were carefully removed from every drum, their roots were examined and platings were made from the lesions, if any, on them. The roots of all plants that were growing in the drums to which O. graminis was added were badly rotted, those of some of the plants in a few of the other drums were, relatively, very mildly affected. The platings from the latter showed that O. graminis was present in several of the lesions. Unfortunately, the cultures were discarded in error before full notes on the distribution of O. graminis were made.

When the grain was ripe, the plants were carefully gathered for examination and evaluation of take-all damage. The standards used were the same as already given in another paper (Angell, 1943), viz., the condition of the roots, the average weight of the plants, the average size of grain and the mean total weight of grain.

(ii) *In* 1942.

During the early part of May, the level of the soil in all the drums was checked and where necessary adjusted by the addition of soil that had been kept in spare drums for the purpose; only a handful or two was required by some, and none at all by others.

During the third week of May, all the drums in the enclosure were sown with twelve grains of Nabawa wheat. No inoculum was used. To each drum was also added the clear solution obtained by adding 10 ml. of water to 1 g. of superphosphate, stirring, allowing the insoluble portion to settle and decanting the supernatant liquid.

During the period 11th August to 8th September, all seedlings in excess of six per drum were removed, and their roots were examined for lesions. Platings were made from the roots of not more than two plants per drum. The growth characters of *O. graminis* were easily recognisable; such cultures were transferred to potato-dextrose agar slopes. Later, they were identified as *O. graminis* by their growth reaction on special media, but it was not practicable, nor for the purpose of this paper was it necessary, further to check identification by the production of perithecia and ascospores in pure culture.

In due course the mature plants were harvested and subsequently disposed of as in the previous year.

(iii) *In* 1943.

Only 16 drums of each series were sown to wheat in 1943. In each series 0·85 g. of sodium nitrate was added to each of four of the drums that were contaminated with *O. graminis* in 1941, and to four others to which only dead mycelium was added in that year. Superphosphate was applied to the drums in the same manner as in 1942. Nabawa wheat was sown on 20th May.

All seedlings in excess of six per drum were removed during the period 10th August to 8th September. The roots were examined for lesions, platings being made from lesioned roots from all plants.

At maturity, all the plants were gathered and set aside until air dry. They were then examined and the extent of take-all damage evaluated according to the standards that were used in the previous year.

3. Results.

(i) *In* 1941.

On account of the influence of some yet undetermined factor, which may have been a suitable degree of soil fertility that enabled them to resist the development of the disease, the plants in the drums to which *O. graminis* was added grew much better than in other years. Throughout the growth period nothing in the appearance of the aerial portions

of the plants indicated that the soil had been contaminated artificially with the organism, and that in consequence many of the roots of every plant that was examined had rotted away.

The differences between the total weights of plants in the uncontaminated and contaminated drums, though significantly in favour of the former, were much less than was usual. Plants grown in the drums to which 250 g. of burnt lime or 250 g. of ground limestone or $61 \cdot 5$ g. magnesium sulphate was added, gave the highest yields and were significantly heavier than those that were grown in the native soil. It will be seen, too, that the mean total weight of grain per drum corresponded well with the mean total weight of the plants. (Table 1.)

The mean grain weight from drums that were contaminated with *O. graminis* was smaller than that from drums that were not contaminated. As was expected from the examination that was made earlier in the season, the roots of the plants in the drums to which *O. graminis* was added were more rotted than those that were produced in soil that was not artificially contaminated.

(ii) *In* 1942.

(a) *White Heads.*—During the growth period preceding jointing, all the seedlings appeared to be growing as uniformly well as is usually expected of a wheat population. As in other experiments that were reported on, or referred to in another paper (Angell, 1943), it was impossible to judge from their general appearance whether or not white heads would develop later. Examination and subsequent plating of the roots of all plants in excess of six per drum showed, however, that lesions were present and that *O. graminis* and other organisms were associated with the lesions. The occurrence of lesions and the isolation of *O. graminis* from the lesions were both significantly associated with the development of white heads. The number of white heads in the uncontaminated drums that contained only the native soil differed significantly at the 1 per cent. level from that in the corresponding drums that were contaminated in 1941. The differences between the responses to the various additions to the soil with the idea of modifying the composition of the exchangeable bases were, however, not significant.

(b) *Weight of Plants and Grain, Size of Grain and Condition of Roots.*—In Table 2 it will be seen that the differences in the weight of the plants, the weight of grain per drum, grain size, and condition of the roots from the contaminated and the uncontaminated drums were all significant. In all these respects, except grain size, the differences between the plants growing in heavily limed and the native soil are also highly significant. Other significant differences are also given in the table.

(iii) *In* 1943.

On reference to Table 3 it will be seen that the plants in the drums that contained only native soil to which neither inoculum nor the chemical compounds listed on page 39 were added in 1941 were much more affected by the disease than were any others. The number of white heads in those drums was significantly greater, and the mean weight of grain was significantly less than in the corresponding drums

TABLE 1.—1941 SEASON.

No.	Chemical	Amt. (g.)	Mean Total Weight per Drum.			Mean Number of Ears per Drum.			Mean Total Weight of Grain (g.).			Mean Grain Weight (g.).			Mean Root Rating.		
			Not Inoc.	Inoc.	Total.	Not Inoc.	Inoc.	Total.	Not Inoc.	Inoc.	Total.	Not Inoc.	Inoc.	Total.	Not Inoc.	Inoc.	Total.
1	Sulphur	2	63·4	61·2	62·3	13·3	14·6	13·9	15·4	14·0	14·7	·050	·048	·049	1·00	1·04	1·02
2	Sodium Sulphate	42	73·2	53·0	63·1	14·8	13·3	14·1	19·4	14·6	17·0	·047	·041	·044	1·03	1·38	1·20
3	Potassium Sulphate	13	53·4	48·0	50·7	10·7	14·1	12·4	13·4	13·9	13·6	·051	·046	·048	1·06	1·26	1·16
4	Magnesium Sulphate	61	96·6	69·1	82·8	16·9	16·0	16·5	21·6	17·3	19·5	·049	·045	·047	1·00	1·05	1·03
5	Calcium Sulphate	22	40·1	42·2	41·1	9·5	12·9	11·2	10·7	12·6	11·6	·050	·042	·046	1·19	1·44	1·31
6	Calcium Sulphate	250	66·8	49·4	58·1	15·9	12·5	14·2	19·3	12·7	16·0	·050	·046	·049	1·07	1·28	1·18
7	Sodium Carbonate	36	69·6	65·7	67·6	13·3	15·1	14·2	18·3	17·0	17·7	·052	·048	·050	1·08	1·23	1·15
8	Potassium Carbonate	13	71·2	62·7	67·0	11·8	14·2	13·0	14·7	16·4	15·5	·053	·047	·050	1·02	1·01	1·02
9	Ground Magnesite	21	57·4	45·8	51·6	14·6	13·1	13·7	19·3	17·3	16·3	·056	·049	·053	1·18	1·79	1·48
10	Ground Magnesite	250	67·3	52·5	59·9	14·1	14·0	14·0	19·2	17·3	18·3	·055	·044	·049	1·28	1·22	1·25
11	Ground Calcium Carbonate	15	54·7	41·6	48·2	10·4	11·1	10·8	12·8	11·1	12·0	·051	·042	·046	1·13	1·37	1·25
12	Ground Calcium Carbonate	250	75·2	65·1	70·1	14·4	15·8	15·1	18·6	17·8	18·2	·051	·044	·048	1·07	1·17	1·12
13	Burnt Lime	15	52·4	39·8	46·1	10·9	10·9	10·9	12·8	11·2	12·0	·054	·049	·052	1·26	1·75	1·51
14	Burnt Lime	250	96·3	82·8	89·5	18·7	17·5	18·1	26·4	26·7	26·5	·046	·038	·042	1·02	1·52	1·27
15	Native Soil (inoc. 1941)		47·3	47·2	47·2	9·5	11·9	11·9	11·1	10·1	10·1	·051	·043	·043	1·08	1·72	1·72
16	Native Soil, nothing added		65·7		47·3	13·3	13·8	9·5	16·9	15·1	11·1	·051	·045	·051	1·10	1·35	1·08
	Mean of All Treatments			55·1		13·3	13·8			15·1	3·26		·045			1·35	

Minimum difference for significance at 1 per cent. between—

Treatments			Not Inoc.	Inoc.	Total.	Not Inoc.	Inoc.	Total.	Not Inoc.	Inoc.	Total.	Not Inoc.	Inoc.	Total.	Not Inoc.	Inoc.	Total.
1–14			15·8	15·8	11·2	3·09	3·09	2·18	5·00	5·00	3·54	·005	·005	·003	35	35	25
1–14 and 15																	
1–14 and 16			12·5	13·7	10·3	2·44	2·68	2·01	3·96	4·33	3·26	·004	·004	·003	28	30	·23
15 and 16																	

SIGNIFICANCE OF GROUP COMPARISONS (Nil treatments excluded).

Chemicals	$P < ·001$	$P < ·001$	$P < ·001$
Inoculation	$P < ·001$	$P < ·001$	$P < ·001$
Chemicals x Inoculation	n.s.	$P < ·05$	$P < ·01$

TABLE 2.—1942 SEASON.

No.	Treatment (Chemical)	Amt. (g.)	Mean Total Weight per Drum (g.)			Mean Total Weight of Grain per Drum (g.)			Mean Grain Weight (g.)			Percentage of Drums with White Head Plants			Mean Root Rating		
			Not Inoc.	Inoc.	Total	Not Inoc.	Inoc.	Total	Not Inoc.	Inoc.	Total	Not Inoc.	Inoc.	Total	Not Inoc.	Inoc.	Total
1	Sulphur	2	19·1	20·0	19·6	4·5	5·8	5·6	·052	·056	·054	11	11	11	1·97	1·86	1·91
2	Sodium Sulphate	42	20·7	23·9	22·3	5·7	6·2	5·9	·051	·055	·053	33	0	16	2·03	1·90	1·97
3	Potassium Sulphate	13	24·6	33·9	29·3	6·4	6·8	6·6	·050	·053	·052	22	33	27	1·37	1·10	1·23
4	Magnesium Sulphate	61	20·6	24·9	22·8	5·2	5·7	5·5	·054	·051	·052	22	33	27	1·98	1·82	1·90
5	Calcium Sulphate	22	23·7	32·1	27·6	3·3	7·6	6·5	·052	·055	·053	44	0	22	1·33	1·02	1·17
6	Calcium Sulphate	250	19·9	18·8	18·8	6·1	5·6	5·9	·051	·055	·053	22	11	16	1·97	1·98	1·98
7	Sodium Carbonate	36	24·7	22·2	23·3	6·4	5·8	6·1	·049	·056	·053	33	0	16	1·82	1·72	1·77
8	Potassium Carbonate	13	21·2	22·6	21·7	6·1	6·1	6·1	·055	·053	·054	11	22	16	1·92	1·93	1·92
9	Ground Magnesite	21	25·3	27·6	26·6	5·3	6·1	5·7	·052	·057	·055	33	22	27	1·91	1·58	1·75
10	Ground Magnesite	250	41·7	51·9	46·8	9·3	11·3	10·3	·048	·057	·052	33	33	33	1·50	1·03	1·26
11	Ground Calcium Carbonate	15	18·3	23·4	20·4	5·6	5·1	5·3	·050	·053	·052	33	11	22	2·13	1·82	1·97
12	Ground Calcium Carbonate	250	39·2	52·9	46·0	7·0	10·7	8·8	·050	·057	·053	55	0	27	1·90	1·02	1·46
13	Burnt Lime	15	26·2	24·7	25·4	6·1	6·7	6·4	·049	·055	·052	33	22	27	1·96	1·41	1·68
14	Burnt Lime	250	82·6	84·2	83·4	18·4	17·0	17·7	·051	·055	·053	11	11	11	1·17	1·19	1·18
15	Native Soil (inoc. 1941)	..	25·8	26·0	26·0	4·7	..	6·4	·045	..	·051	61	..	11	2·19	1·67	1·67
16	Native Soil, nothing added	..	29·5	32·6	25·8	6·9	7·5	4·7	·051	·055	·045	30	15	61	1·81	1·54	2·19
	Mean of All Treatments	..		32·6			7·5			·055			15			1·54	

Minimum difference for significance at 1 per cent. between—

Treatments	Not Inoc.	Inoc.	Total	Not Inoc.	Inoc.	Total	Not Inoc.	Inoc.	Total				Not Inoc.	Inoc.	Total
1-14	10·9	10·9	7·7	2·6	2·6	1·8	·009	·009	·006				·47	·47	·33
1-14 and 15															
1-14 and 16	8·6	9·4	7·1	2·1	2·3	1·7	·007	·007	·006				·37	·41	·31
15 and 16															·31

SIGNIFICANCE OF GROUP COMPARISONS (Nil treatments excluded).

Chemicals	P < ·001	P < ·001	P < ·001	n.s.	
Inoculation	P < ·001	P < ·001	P < ·001	P < ·001	
Chemicals x Inoculation	n.s.	n.s.	n.s.	n.s.	

Chemicals	..	P < ·001
Inoculation	..	P < ·001
Chemicals x Inoculation	..	P < ·05

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containing soil that was contaminated in 1941, but there were no significant differences between the total weight of the plants, the total weight of grain, or the condition of the roots in the two series. The ratio of the mean weight of grain in the two series corresponded to the ratio of the total weight of grain, but owing to lower precision the latter was not significant.

The plants from the drums to which the organism and chemicals were added in 1941 weighed more and produced more grain than did those from the corresponding uncontaminated controls. This was the second reversal in the relative magnitude of the yields, the order being the same as in 1941 and the reverse of that of 1942.

The total weight of the plants and the total weight of the grain borne by the plants in the drums to which sodium nitrate was added in 1943 were greater than in the corresponding controls, but there was no difference between the size of the grain, the condition of the roots, or the number of drums with white-headed plants in the two series.

There was no evidence of any significant differences between the effects of any of the other added chemicals on white heads, on plants with pinched grain, or on the condition of the roots. The addition to the soil of the larger quantities (250 g.) of burnt lime, ground limestone, and ground magnesite was associated with greater total weight of the plants and greater total weight of grain than was any other added chemical, burnt lime being outstanding.

4. Discussion.

Under the conditions of the experiments that have been made from 1938 to the present time, more or less restricted development of the wheat plant due to indifferent nutrition during the vegetative stage was usually later associated with marked symptoms of take-all, including the production of small crops of pinched grain. Premature death of plants sometimes occurred. Well developed plants such as were produced in the first year from a new lot of soil, or in the first two years in heavily limed soil, or in the second year of the experiment in drums in which the first year's crop failed to develop well on account of artificial contamination with *O. graminis*, resisted the disease satisfactorily. Good crops from such plants were the rule.

The general trend of the results of all the experiments was that the deliberate introduction of the organism into some drums in the first year did not result in a poor, but a comparatively good crop, in the second season. On the contrary, in the drums to which cultures of the organism were not added at any time, a poor crop was obtained in the second season. If take-all damage were dependent on the concentration of the organism, the results should have been the reverse. The second year's experimental results were, in the writer's opinion, due mainly to differences in the amount of plant food available. The alternative, that the concentration of the organism in the second year was less in the drums into which it was introduced a year previously and greater in other drums in which it may have been present initially or subsequently introduced by chance, and that the second year's crop was influenced mainly by this change in concentration, does not appear to be a reasonable assumption.

TABLE 3.—1943 SEASON.

No.	Treatment (Chemical)	Amt. (g.)	Mean Total Weight per Drum (g.)			Mean Total Weight of Grain per Drum (g.)			Mean Grain Weight (c.)			Percentage of Drums with White Head Plants.			Mean Root Rating.		
			Not Inoc.	Inoc.	Total.	Not Inoc.	Inoc.	Total.	Not Inoc.	Inoc.	Total.	Not Inoc.	Inoc.	Total.	Not Inoc.	Inoc.	Total.
1	Sulphur	2	37·3	29·9	33·6	10·1	9·0	9·5	·054	·052	·053	25	0	12	1·84	2·00	1·92
2	Sodium Sulphate	42	29·9	26·6	28·2	8·1	9·8	9·0	·052	·054	·053	37	0	19	1·89	1·81	1·85
3	Potassium Sulphate	13	41·2	30·2	35·7	10·6	8·1	9·3	·054	·056	·055	12	0	6	1·87	1·61	1·69
4	Magnesium Sulphate	61	31·5	25·5	28·5	11·1	7·9	9·5	·046	·051	·049	50	0	25	2·04	1·69	1·86
5	Calcium Sulphate	22	32·9	33·2	33·0	5·6	8·9	7·2	·053	·054	·054	25	0	12	2·03	1·87	1·95
6	Calcium Sulphate	250	34·6	23·3	28·9	8·1	6·5	7·3	·054	·056	·055	12	12	12	1·91	1·98	1·94
7	Sodium Carbonate	36	44·4	35·9	40·7	13·5	11·6	12·6	·057	·058	·058	0	25	31	2·05	1·64	1·84
8	Potassium Carbonate	13	36·9	29·0	32·9	9·3	9·0	9·1	·052	·053	·053	37	12	19	2·09	2·14	2·11
9	Ground Magnesite	21	45·9	38·2	42·0	12·3	10·0	11·1	·055	·053	·054	25	25	31	2·04	2·00	2·02
10	Ground Magnesite	250	52·9	54·2	53·5	17·8	15·1	16·4	·052	·049	·051	25	37	31	1·91	1·62	1·77
11	Ground Calcium Carbonate	15	33·9	30·3	32·1	8·3	8·8	8·6	·054	·054	·054	0	0	0	1·64	1·93	1·79
12	Ground Calcium Carbonate	250	55·9	54·4	57·0	14·5	15·5	15·0	·053	·054	·053	25	37	31	1·40	1·44	1·42
13	Burnt Lime	15	48·1	39·4	43·7	12·8	9·8	11·3	·055	·054	·054	12	25	19	1·57	1·59	1·58
14	Burnt Lime	250	84·3	74·5	79·4	21·1	20·3	20·7	·052	·053	·053	0	0		1·99	2·04	2·01
15	Native Soil (inoc. 1941)			36·4	36·4		10·3	10·3		·054	·054		0			2·07	
16	Native Soil, nothing added		32·5		32·5	9·3		9·3	·051			41			1·82		
	Sodium Nitrate on chemical treatments		47·8	38·0	42·8	12·6	11·0	11·8	·054	·053	·054	12	18	15	1·84	1·87	1·85
	No Nitrate on chemical treatments		39·8	36·9	38·3	10·7	10·4	10·5	·053	·054	·054	9	23	16	1·91	1·75	1·83

Minimum difference for significance at 1 per cent. between—

Treatments			Not Inoc.	Inoc.	Total.	Not Inoc.	Inoc.	Total.	Not Inoc.	Inoc.	Total.				Not Inoc.	Inoc.	Total.
1–14			14·0	14·0	9·9	4·7	4·7	3·3	·005	·005	·004				·56	·56	·40
1–14 and 15																	
1–14 and 16			11·1	12·1	9·1	3·7	4·0	3·1	·004	·004	·003				·45	·49	·37
15 and 16																	

SIGNIFICANCE OF GROUP COMPARISONS (Native Soil excluded).

	Not Inoc.	Inoc.	Total.		P < ·01
Chemicals	P < ·001	P < ·001	P < ·001	P = ·001	
Inoculation	P < ·001	P < ·001	P = ·05		
Chemicals x Inoculation	n.s.	n.s.	n.s.	n.s.	n.s.
Sodium Nitrate	P < ·01	P < ·01	n.s.	P < ·01	n.s.
Sodium Nitrate x Inoculation	P < ·05	P < ·05	n.s.	n.s.	n.s.

In the wheat-growing areas, cropping with wheat year after year produces in time the same type of result as was obtained in the second and succeeding years in the control soil used in these experiments. The supply of plant food that is available becomes unbalanced by repeated cropping, the plant fails to grow satisfactorily, and *O. graminis* develops rapidly at the host's expense during or after jointing. In good farm practice, conservation of plant food is achieved by rotation of crops or fallowing, or the application of manure; take-all seldom occurs under such conditions unless there is some limiting factor.

The form in which nitrogenous fertilizers are added might prove to be more important than the amount of nitrogen in them. The organic forms of nitrogen may exercise more influence on the course of the disease than the inorganic. In this experiment, the addition of nitrate nitrogen, although promoting growth and yield, did not appear to control take-all. Experiments to determine the influence of some nitrogenous fertilizers are in progress.

In none of the experiments have very significant differences been noted as a result of the addition of compounds that result in modification of the exchangeable bases in the soil, burnt lime being a notable exception. The mode of action of large amounts of partly carbonated burnt lime in the soil, resulting in the release of plant food for successive crops of wheat and the control of take-all, is not known. None of the work that has been reported by soil scientists appears to be applicable to this particular case.

5. Acknowledgments.

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Note on the Establishment of *Phalaris tuberosa* in 1944 at Canberra, A.C.T.

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In April 1944, a pasture consisting of *Phalaris tuberosa* and sub-terranean clover was sown at the Dickson Experiment Farm, Canberra, to test subterranean clover strains under grazing conditions. The season was abnormally dry, but in spite of this a satisfactory stand of *Phalaris* was obtained.

The total rainfall during 1943 was 23·62 inches, of which 10·22 inches fell between 1st September and 31st December. During 1944, the rainfall from 1st January until the seed was sown on 5th April was only 1·72 inches, and from 6th April to 20th November, 5·52 inches, of which falls of 20 points or more accounted for only 3·94 inches.

The site chosen for the experiment was three acres of an old culti-vation paddock which was last cropped in 1939 and had since then been allowed to revert to pasture. The area was grazed bare before being ploughed to a depth of 6 inches with the mouldboard plough in mid-September 1943. The ground was in ideal condition for ploughing, and the sod was turned well underneath. Only two major cultivations were required subsequent to ploughing, these being done with the " Sunder-cut " (disc cultivator) to a depth of 5 inches on 10th December, 1943 and to a depth of 4 inches on 4th February, 1944. In addition, the land was harrowed after ploughing and prior to seeding. At no time was the surface soil broken down beyond a coarse, lumpy condition.

The *Phalaris* seed was sown at a depth of approximately $1\frac{1}{2}$ inches with the disc drill on 5th April, 1944 at the rate of 3 lb. per acre. The seed was mixed with 18 per cent. superphosphate which was sown at the rate of 1 cwt. per acre. By sowing at a depth of $1\frac{1}{2}$ inches the seed was placed just above moist soil, and the light falls of rain which occurred in April and May were sufficient to cause germination and brairding. The subterranean clover plots were sown by hand on 6th April, 1944, and the whole area harrowed.

An estimate of the establishment of *Phalaris* was made on 20th November, 1944. A stick 10 links long marked in $\frac{1}{2}$-link lengths was placed beside the *Phalaris* drill rows in two random positions on each subterranean clover plot, making a total of 90 positions. The presence or absence of *Phalaris* plants in each $\frac{1}{2}$-inch length was noted. From this information, the presence or absence of *Phalaris* plants was subse-quently determined in lengths of 1, $1\frac{1}{2}$, and 2 links. At least one plant was present in 45·90 ± 5·40 per cent. of $\frac{1}{2}$-link lengths, 64·78 ± 6·04 per cent. of 1-link lengths, 76·85 ± 5·68 per cent. of $1\frac{1}{2}$-link lengths, and 82·66 ± 5·64 per cent. of 2-link lengths.

Assuming that one well established plant per square link would eventually provide an adequate proportion of *Phalaris* in the mixed pasture, the stand obtained in this experiment can be regarded as very satisfactory and indicative of the ability of *Phalaris* to establish under very adverse climatic conditions providing the seed bed has been suitably prepared.

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The Relationship between Necrosis and Resistance to Virus Y in the Potato.

1. Greenhouse Results

By E. M. Hutton, M.Sc.,* and J. G. Bald, M.Agr.Sc., Ph.D.*

Summary.

1. Hypersensitive necrotic reactions to virus Y are described in the seedling progeny of some potato crosses.

2. It is considered that these reactions will provide field resistance to virus Y in the same way that Epicure is protected from virus X and A.

3. It is postulated that most potato crosses will give a small percentage of the hypersensitive types described.

4. It appears that the hypersensitive reactions to virus Y are heritable but their mode of inheritance is not understood.

1. Introduction.

The potato breeding programme at Canberra has been designed for the production of hybrids resistant to viruses and of good agronomic type. Since resistance to viruses A and X is already present in a number of commercial varieties (Cockerham, 1943a), it was decided to examine the possibility of obtaining resistance to virus Y and leaf roll. Up to the present most progress has been made in developing hybrids resistant to virus Y.

Previous research on resistance to virus Y has not been encouraging. In a review of potato virus diseases in England, Bawden (1942) remarked that although hypersensitivity to virus X has been found in King Edward and Epicure no varieties have been found with this type of reaction to Y. Recently Cockerham (1943b) summed up the position in the British Isles by stating that some form of protection can be obtained against most of the important potato viruses but not as yet against virus Y.

That some degree of resistance to virus Y is present in certain potato varieties is claimed by a few workers. Chippewa, Katahdin, Sebago, and Russet Burbank have been found by Jones of the State College of Washington (1941) to possess considerable resistance to field infection by virus Y. Bald and Pugsley (1941) reported that the Australian Snowflake reacts with great severity to virus Y and infected plants yield very little. Diseased plants in seed stock have few descendants and the incidence of the disease is rapidly reduced in successive vegetative generations, unless the current rate of spread is high. Salaman (1943) stated that *Solanum Rybinii* and a few other wild species may be resistant to virus Y and that a Chilean variety of Müller's is reported to be immune.

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2. The Problem of Developing Resistance to Virus Y.

In attempting to develop hybrids resistant to virus Y it was postulated that the necrotic reaction of varieties like Snowflake could be increased by intercrossing to the point where seedlings became hypersensitive, as Epicure is to virus X and A. This approach seemed to have more chance of success than breeding for immunity or no-reaction to virus Y. Further it was thought that genes conditioning such a reaction would most likely be found in old varieties which were still being grown and which showed only a small degree of virus degeneration. For this reason Snowflake and Brown's River were chosen as parents. Both of these varieties are male sterile so the American variety Katahdin was used as the pollen parent. This variety, apart from certain valuable agronomic characters, undoubtedly possesses certain genes governing the necrotic reaction to virus Y.

Two crosses—Snowflake x Katahdin and Brown's River x Katahdin —were made in the early summer of 1941. At the same time the variety Katahdin was selfed. Seed was gathered from the crosses and the selfed Katahdin during February, 1942, and the seedling were raised during the winter in the greenhouse. Small tubers were harvested from the seedlings in July, 1942, and these were planted in the greenhouse during October, 1942. Inoculation and observation of the hybrids and selfed Katahdin progeny took place during November, 1942.

3. Reaction of the Parent Varieties to Virus Y.

Snowflake, Brown's River, and Katahdin have been inoculated with virus Y at various times by means of the aphid vector *Myzus persicae*, by grafting, and by rubbing with a spatula and carborundum. The reactions of these varieties to virus Y has also been observed under various conditions in the field. In one instance mechanical inoculation of the three varieties under rapid growing conditions in the early summer gave the following reactions which are fairly typical:—

(i) *Snowflake.*—The inoculated leaves became yellow and necrotic and finally dropped off. Six weeks from the time of inoculation a fairly severe systemic necrosis had developed and a mottle was present on the upper leaves. Under other conditions varying degrees of necrosis may develop. Necrosis developing on the stem and young leaves may sometimes kill the growing point.

(ii) *Brown's River.*—The inoculated leaves showed no necrosis but gradually yellowed and then fell off. Six weeks from inoculation the upper leaves were slightly rugose and had developed a mild mottle. The period between inoculation and the first appearance of systemic symptoms was more prolonged than on the other two varieties. No systemic necrotic symptoms appeared. Under cooler conditions the mottle may be of medium intensity and the rugosity more pronounced.

(iii) *Katahdin.*—The inoculated leaves became yellow and necrotic and finally dropped off. Six weeks after inoculation systemic necrotic areas had appeared in several places on the plant and the younger leaves were mottled and rugose.

In other instances streaking of the stem has been the most prominent symptom. The reactions of Snowflake and Katahdin to virus Y are similar but that of Snowflake is the more severe.

Inoculation of these varieties with Y by infectious aphids (*Myzus persicae*) may result in a severer reaction than that obtained from mechanical inoculation, particularly by Snowflake, although the types of reaction in each case are much the same. In addition the appearance of systemic symptoms is more rapid.

Grafting of Epicure scions containing virus Y on to suitable stocks of these three varieties resulted in three different types of reaction. In Snowflake a total necrotic collapse of the stock resulted in two weeks, whereas in Brown's River a reaction closely approaching top necrosis was produced. In Katahdin, apart from a few scattered necrotic patches on the plant no well defined reaction resulted in two weeks, although severe symptoms developed later.

In the field, current season symptoms on Katahdin and Snowflake are typically necrotic. Current season symptoms on Brown's River have not been observed. The symptoms in succeeding years both in greenhouse and field are similar.

4. Reactions to Virus Y Observed in Seedlings.

Seedling reactions to inoculation were observed in most cases during the spring and early summer as under these conditions a truer picture is obtained than in the hotter or colder periods of the year. Reactions could be classified into three distinct groups: (i) A mild mottle without rugosity. Sometimes plants in this group appeared to give no reaction. (ii) Typical rugose mosaic with or without some systemic necrosis. (iii) Severe systemic necrosis without a mottle, which results in the complete collapse of the plant.

Table 1 shows the proportions of seedlings in the crosses and inbred Katahdin which gave the various reactions.

TABLE 1.—REACTIONS OF POTATO SEEDLINGS TO VIRUS Y.

	Mild Mottle.	Rugose Mosaic.	Necrotic Collapse.	Total.
Snowflake x Katahdin	189	55	37	281
Brown's River x Katahdin ..	183	80	51	334
Katahdin selfed	45	16	13	74

These results were promising, approximately one-sixth of the material handled possessing a reaction to virus Y severe enough to warrant more intense study. That the selection of the parents was justified was borne out to some extent by the reactions to virus Y observed on seedlings raised from selfed Bismarck and Carman seed. Bismarck reacts to infection with virus Y almost without necrosis and although Carman may react with leaf-drop streak, the necrosis on the lower leaves is often ill defined even at the first appearance of symptoms. Of 94 inbred Bismarck seedlings none showed necrotic collapse and all possessed a distinct mottle and mild rugosity which is characteristic of Bismarck itself. Among 340 inbred Carman seedlings 316 had a distinct mottle with some rugosity and only 26 were completely killed by the virus.

Probably a certain percentage of necrotic collapse to virus Y would be found in most potato crosses, but the proportion of hybrids possessing this reaction would be higher in some crosses than in others. This aspect of the problem needs further work.

5. The Types of Necrotic Reaction Present in the Selected Seedlings.

It was realized that the necrosis obtained in many of the seedlings could be atypical since they were grown from small tubers and were not so vigorous as plants from normal seed tubers. Part of all seedling lines which gave a necrotic collapse to virus Y were grown on again to form normal size tubers in the second year. The reactions of plants grown from such tubers have been studied under various conditions in comparison with the parent varieties and other varieties commonly grown in Australia.

It was found that a number of the hybrids originally selected did not show a pure necrotic reaction to virus Y but that a mottle accompanied the necrosis. Such hybrids when well-grown before inoculation gave the typical leaf-drop streak symptoms of virus Y and were consequently discarded. Other hybrids developed a fairly severe systemic necrosis and mottle without leaf-drop streak symptoms. These also were rejected. All but twelve of the original selections were eventually eliminated. Tubers from the rejected seedlings giving both types of reaction produced dwarfed plants with severe rugose mosaic similar to those raised from the tubers of infected Snowflake plants.

Among the group of hybrids retained were three types of necrotic action which appear promising as a basis for resistance to virus Y. In all cases the necrotic reaction is a pure one unaccompanied by mottling of any kind. Plates 7, 8, and 9, showing hybrids 48, 106, and 404, illustrate these necrotic reactions during early summer. The plants at inoculation were approximately 2 feet high and were growing vigorously. The photographs of the plants were taken four weeks after inoculation and those of the leaves ten days after inoculation. Hybrid 48 after inoculation becomes severely necrotic and develops a severe leaf-drop streak, which results in the total collapse of the plant within a month. Hybrid 106 rapidly develops top necrosis which finally results in the collapse of the growing point. With both hybrids, the reaction on the inoculated leaves after ten days is severe, resulting in severe necrosis, yellowing, and then leaf-drop. With 404 relatively small localized necrotic lesions appear rapidly on the inoculated leaf and may or may not be followed by leaf-drop. In some 404 plants the infection remains entirely localized in the inoculated leaves whereas in others dropping of the inoculated leaf is followed by stem necrosis for several inches from the point of leaf abscission, no other symptoms subsequently appearing. In other 404 plants again, relatively mild systemic necrosis develops which results in the death of the infected leaflets. Symptoms then develop no further. Snowflake, Brown's River, and Katahdin plants inoculated at the same stage of growth give much slower and less definite reactions on the inoculated leaves, and the development of systemic symptoms takes four to six weeks longer than on the hybrids mentioned.

That the three types of necrotic reaction present in the hybrids constitute a protective barrier against virus Y under greenhouse conditions is proved by the fact that tubers from infected plants produce either healthy plants or small severely necrosed plants which die rapidly soon after emergence from the soil.

A further encouraging fact is that these hypersensitive hybrids when grafted with an Epicure scion containing virus Y react with a severe top necrosis in fourteen days.

6. The Effect of Growth Conditions on Reactions to Virus Y.

It was found that, during the winter in the greenhouse when growing conditions are relatively slow, many hybrids gave reactions to Y localized to the inoculated leaves. Tubers produced by the inoculated plants gave plants free of virus Y. That false results can be obtained under winter conditions was proved when these hybrids were retested under the quick-growing conditions of early summer, many of them then being discarded because of unsatisfactory reactions.

Atypical results can also be obtained if very young or weakly growing plants are used for testing. With such plants the virus reaction is always much more severe and mottle symptoms can be suppressed.

7. Possibility of the Necrotic Reaction being a Heritable Character.

Selfing or intercrossing the valuable hypersensitive hybrids results in at least one-third of the seedling progeny having promising reactions to virus Y. On the other hand crossing hypersensitive hybrids with potato varieties possessing the typical rugose mosaic reaction to virus Y results in approximately 5 per cent. of the seedling progeny showing valuable necrotic reactions to this virus.

Much more work needs to be done on this aspect of the problem to elucidate the genetical factors involved. All that can be said is that these necrotic reactions are not fortuitous and can be recovered in crosses, the percentage recovered depending on the reactions of the parents.

8. The Relationship between Greenhouse and Field Infection.

Research on this aspect of the problem is in progress and results to date appear promising. Aphis infection of hypersensitive hybrids gives, if anything, a more rapid and severe necrotic reaction than that obtained in the greenhouse.

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The Keeping Quality of Tinned Butter.

By E. G. Pont, M.Sc.Agr.*

Summary.

Tinned butter has shown pronounced liability to deterioration when used by the Services in hot climates. The principal defects have been rancid, cheesy, and putrid flavours due to bacterial activity, and the objectionable physical condition consequent on melting.

In experiments dealing with the keeping quality of butter at summer room temperatures, bacterial defects were controlled by the use of boric acid or by a process of vacuum working combined in each case with salt concentrations ranging from 2·5 to 3 per cent. or more. Under these conditions, however, quality was still seriously impaired by tallowy flavours due to fat oxidation.

The rate and extent of fat oxidation in tinned butter was not affected by a 15°F. increase in pasteurization temperature or by reduction of air content in vacuum processing. Copper contamination, even in the low range of 0·06 to 0·17 p.p.m. was important, being correlated with peroxide and aldehyde values and final grades after holding three months.

Ethyl gallate in a concentration of 0·02 per cent. was found to have marked anti-oxidant properties in pure butterfat. Tested in tinned butter it showed a similar anti-oxidant effect judged by low peroxide values and the absence of pronounced tallowy characteristics, but deleterious flavour changes still occurred.

It was concluded that the essentially perishable nature of butter could not be sufficiently modified in the directions attempted to render it suitable as a tinned product for Service use.

1. Introduction.

This paper reports the results of experiments which were designed to improve the keeping quality of tinned butter supplied to the Services in desert and tropical areas. Although some progress was made it was impossible to avoid the conclusion that tinned butter was an unsuitable product for such use. The problems of its storage and distribution in hot climates were solved ultimately only by the development in this laboratory of a high melting-point butterfat spread. The results of the experiments with tinned butter are published because they illustrate the type and degree of deterioration to which this product is liable as well as having some bearing on the keeping quality of butter in general.

The impossibility, under Service conditions, of discrimination in the treatment of individual food products necessitated the handling and storage of tinned butter in the same manner as foods of a non-perishable nature. In these circumstances severe deterioration was constantly encountered. Reports from Service areas and examination of samples from defective consignments indicated that rancid, cheesy, and putrid defects predominated. Where the butter had been subjected to temperatures above its melting point, deterioration was rapid and severe. Under these conditions butterfat and serum separate into more or less well defined layers and the normal physical restrictions on bacterial activity are no longer operative. The resultant combination of both objectionable physical condition and flavour rendered the butter unfit for consumption.

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Australian tinned butter usually contains about 2 per cent. of salt in contrast to 1 to 1·5 per cent. in the normal domestic and export product. Butter from areas known to produce a harder type of fat is selected if possible, for tinning purposes, but this and the addition of extra salt are the only provisions usually made for increased keeping quality, and resistance to unfavourable temperatures. Where export regulations permit its use, however, 0·25 per cent. of a borate preservative is added. In some instances the additional ingredients are added during manufacture and the butter immediately tinned. Usually, however, bulk butter is selected for tinning and re-worked with the calculated quantity of extra salt and preservative if the latter is required.

2. The Effect of Salt, Boric Acid, and Re-working on the Keeping Quality of Butter at Room Temperature.

The flavour defects referred to as being of common occurrence in tinned butter are caused by bacterial activity. It appeared likely that improvement in keeping quality could be effected by the adoption of more efficient measures to control bacterial development. With this end in view, a preliminary small-scale experiment was planned in order to test the effectiveness of high salt concentrations and boric acid in controlling bacterial defects. The potentially harmful effects of re-working butter are well known; it appeared desirable, therefore, to determine whether the incorporation of extra salt in the manner normally employed in tinning butter would compensate for the unfavourable effect on keeping quality. Although boric acid has been used extensively as a butter and margarine preservative, reports in the literature are conflicting in regard to its efficiency. O'Callaghan (1912) has reported experiments in which butters containing 0·5 per cent. of boric acid were 1 to 3 points superior in grade, compared with control butters kept for several weeks at cool-room temperatures. More recently, however, a report of New Zealand experiments (Anon, 1925) indicated that a "boron compound" in concentrations ranging from 0·1 to 1·0 per cent. showed to little or no advantage as a preservative in butter at cold store and room temperatures over a period of some weeks. Enquiry into the usefulness of boric acid was made in view of the inconclusive nature of this and other evidence.

Butter in the preliminary experiment was made in 1½ to 2 lb. lots from batch-pasteurized sweet cream. Some difficulty was experienced in controlling salt and moisture concentrations, and figures for salt, for the purposes of comparison, have been calculated on a 16 per cent. moisture basis. The cream subsequent to pasteurization was inoculated with crude cultures from rancid tinned butter. The butter was held for six weeks at room temperatures which varied from about 18° to 28°C. Total bacterial counts were determined at intervals and the results together with details of manufacture and treatment of the butters are given in Table 1. The pH of various samples of butter serum varied between 6·8 and 7·0; boric acid was added as a mixture of equal parts of the acid and its equivalent in sodium borate.

With the small scale of manufacture the texture of the butter in this series was inferior to that of commercially made butter. Only tentative conclusions, therefore, could be derived. The harmful effects of re-working were very obvious; in spite of the addition of 0·75 per cent.

additional salt, re-worked butter deteriorated very badly and had very much higher bacterial counts compared with the control. The preservative effect of boric acid was clearly demonstrated. Even in re-worked butter, no bacterial development occurred over the six weeks' period, and this butter was finally easily the best of the series. With inferior texture even a salt concentration of 3·45 per cent. was insufficient to prevent slow bacterial growth and the development of rancidity. Where bacterial growth was restrained, tallowy oxidative changes occurred.

TABLE 1.—EFFECTS OF RE-WORKING OF BUTTER, BORIC ACID, AND HIGH SALTING ON BACTERIAL DEVELOPMENT AT ROOM TEMPERATURE.

Treatment of Butter.	Salt.	Total Bacterial Counts per cc.			Final Flavour.
		Initial.	2 weeks.	6 weeks.	
Control	% 2·05	20,000	1,200,000	800,000	Very rancid
Original salt concentration 2·05 per cent. Re-worked after 3 days with 0·75 per cent. extra salt	2·80	20,000	1,536,000	24,000,000	Extremely rancid and objectionable
Original salt concentration same as control. Re-worked after 3 days with 0·65 per cent. extra salt and 0·25 per cent. boric acid	2·7	20,000	30,000	10,000	Oily and tallowy
Salt added to cream and butter washed at grain stage with 11 per cent. brine	1·7	13,000	1,820,000	1,200,000	Very rancid
Salted in normal manner, but high concentration aimed at	3·45	31,000	362,000	370,000	Metallic and tallowy. Rancid tendency

3. The Effects of Vacuum Processing, Boric Acid, and Excessive Pasteurization Temperatures on the Keeping Quality of Tinned Butter.

Subsequently, Sutton (1942) reported the results of contemporary experiments in which butter was salted by washing at the grain stage with saturated brine, re-worked in an Abel vacuum worker*, and tinned under commercial conditions. The final salt concentrations were from 2·4 to 2·9 per cent. After several months storage at room temperature no bacterial defects were evident, but pronounced tallowy flavours made the butter unpalatable. A commercial scale experiment was accordingly arranged in which the relative merits of boric acid and vacuum processing on the keeping quality of tinned butter could be assessed. As a

* In the Abel vacuum worker (Abel, 1929), butter is beaten in a jacketed metal compartment under vacuum by metal blades revolving at high speed. The process is claimed to produce a butter with a fine moisture dispersion and low air content.

means of controlling oxidized and tallowy flavours the possibilities of excessive pasteurization temperatures, such as would lead to the production of cooked flavours, were also investigated. Overheating of milk and the consequent formation of a cooked flavour are known to inhibit the development of oxidized flavours in this product (Brown and Thurston, 1940).

In this experiment butter was manufactured on a commercial scale from choicest quality cream pasteurized in a Bell volatilizer (Hunziker, 1940). The critical temperature for the production of cooked flavours with this particular unit, according to previous experience was 195°-200°F. Half the cream was treated at 192°F. and the other half at temperatures which through difficulties of control varied between 200°F. and 214°F. Most of the cream was heated to about 208°F. In order to intensify the effect of excessive heat treatment the butter was worked without washing and moisture was made up where necessary by adding buttermilk. Subsequent treatments were varied to test the relative effects of vacuum processing and boric acid, combined and individually, on keeping quality. The butter was packed and sealed in 1-lb. tins and stored at room temperature for three months. This ranged between 16°C. and 22°C., but for a period of five days in each month the tins were placed in a 30°C. incubator. The butters were graded at the beginning and end of the storage period. A summary of the results is presented in Table 2.

TABLE 2.—THE EFFECTS OF VACUUM PROCESSING, BORIC ACID, AND INCREASED PASTEURIZATION TEMPERATURES UPON KEEPING QUALITY OF TINNED BUTTER.

Treatment.	Pasteurization Temperature.	Salt.	Boric Acid.	Grade Scores and Comments.	
				Initial.	3 months.
Normal ..	Deg. F. 192	% 3·02	% ..	Common choicest— 93 pts.	Stale, cheesy—84 pts.
		2·53	0·12	Common choicest— 93 pts.	Stale, tallowy—86 pts.
	208	3·37	..	Very slight processed flavour—93 pts.	Stale, tallowy and soda flavour—83 pts.
		2·75	0·14	Very slight processed flavour—93 pts.	Stale, tallowy—85 pts.
Vacuum processed	192	3·02	..	Common choicest— 93 pts.	Stale, cheesy—87 pts.
		2·53	0·12	Common choicest— 93 pts.	Stale tallowy—86½ pts.
	208	3·37	..	Very slight processed flavour—93 pts.	Stale tallowy slight soda aroma—85 pts.
		2·75	0·14	Very slight processed flavour—93 pts.	Stale tallowy—85½ pts.

The conditions of this experiment were not entirely satisfactory. The amount of cooked flavour produced was not so great as had been expected, only a slight processed flavour being discernible in the fresh butter. The final concentrations of salt and boric acid varied considerably from those aimed at especially in the case of boric acid, the concentration of which, by analysis, was only 0·12 per cent. instead of 0·25 per cent. The texture of the normally worked butters was faulty, excessive free moisture and mottling being evident in many of the tins opened. The condition of the vacuum processed butters was not entirely characteristic of butter subjected to this treatment, free moisture again being present. These factors, however, were irrelevant to the main conclusion derived from the results, that elimination of bacterial defects only brought into strong emphasis tallowy flavours due to fat oxidation. The use of both boric acid and vacuum processing resulted in a substantial increase in grade score, but the best of butters finally were only a mediocre second grade, and the tallowy characteristics were very pronounced. Bacterial counts on the fresh butters ranged from 5,000 to 45,000 per cc. In both the normally worked and the vacuum processed series counts increased on storage to a level of about 300,000 per cc. In the presence of boric acid there was a consistent decrease. The use of higher pasteurization temperatures and non-washing of butter gave no indication that any improvement could be expected in this respect even over a more extreme range, butters from this series grading lower than in the normally pasteurized series. The results of air determinations and peroxide and fat aldehyde values indicated that the rate and degree of oxidation was not influenced by the air content, and inferentially the oxygen content, of the butters. Air determinations carried out according to the method demonstrated by Guthrie (1930) gave values varying from 3·72 to 6·52 per cent. by volume at N.T.P. with normally worked samples, and 0·35 to 1·38 per cent. in the case of the vacuum processed series. Copper contents determined by the method of Moir and Andrews (1940) showed a well defined relationship to peroxide and aldehyde values and appear to have exerted a definite controlling effect upon final quality and grades, particularly in the vacuum processed butters. As the values were already low, ranging between 0·06 and 0·17 p.p.m., the possibilities of substantial improvement by measures to further reduce copper contamination were not bright.

4. Bacterial Defects in Brine-Washed and Vacuum Processed Butter : Preliminary Experiments with Anti-Oxidants.

In the previous experiment vacuum processing resulted in an increase in keeping quality, but the effect was not associated with a comparable decrease in bacterial counts. In spite of this anomaly, which was possibly associated with irregular salting, inferior texture, and the absence of detrimental types of organisms, it was decided to rely on vacuum processing for the control of bacterial defects in subsequent experiments. This course was taken in view of the satisfactory results reported by Sutton (1942). Possible objections to the use of boric acid in butter and the desirability of limiting the number of experimental treatments was also taken into consideration.

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The results of further contemporary experiments reported by Randall and Sutton (1942) indicated that the use of caramelized skim milk in butter manufacture and the vacuum sealing of tins were ineffective in controlling tallowy flavours. Success in this respect appeared most likely of achievement through the use of more specific anti-oxidants. A preliminary experiment was carried out in which several substances suggested in the literature or used commercially for the stabilization of edible fats were tested. Butter in this experiment was made in 10 lb. lots from commercial pasteurized cream. It was salted by brine washing and processed after manufacture in a vacuum worker. Although the texture was satisfactory and the salt efficiently distributed, the significance of the experiment was obscured by the development of rancid flavours. This was traced to the fact that the cream was initially badly contaminated in one series and in another there was active development of lipolytic yeasts in spite of low initial counts. The salt concentrations ranged from 2·23 to 2·78 per cent on a 16 per cent. moisture basis. The results indicated the desirability of higher salt concentrations than these for adequate control of bacterial defects and the continued need for rigid sanitary precautions to reduce contamination to a minimum.

The anti-oxidants tested were an alcoholic extract of oat flour and sugar, a commercial anti-oxidant referred to as No. 221, and an ether extract of Kamala. The use of Kamala as an anti-oxidant in ghee has been reported on favourably by Govindarajan and Banerjee (1940). The general appearance of rancid flavours obscured flavour judgments in respect of tallowiness but fat peroxide values were significant. These are given in Table 3.

TABLE 3.—FAT PEROXIDE VALUES FROM TINNED BUTTER CONTAINING ANTI-OXIDANTS. SIX WEEKS' STORAGE AT ROOM TEMPERATURE.

Treatment.	Lea Peroxide Values	
	Original.	6 weeks.
Control	0·17	0·67
Oat flour and sugar extract added to butter	0·09	0·65
No. 221 added to butter	0·15	0·74
Kamala extract added to butter	0·05	0·13

The low peroxide value obtained with the Kamala extract suggested that this or some more suitable substance might be effective in controlling the tallowy flavours, which occurred in the absence of bacterial defects.

5. The Use of Ethyl Gallate as an Anti-Oxidant in Tinned Butter.

Kamala extract imparted a deep unnatural yellow colour to butter, and apart from its apparent anti-oxidant effects, it possessed characteristics which rendered undesirable its use in butter. Ethyl gallate was suggested as having more potential advantages, and a test of the relative anti-oxidant effects of the two substances was carried out. In this experiment the Kamala extract and ethyl gallate were added to pure butterfat, both in the absence and presence of added copper in the form

of copper stearate. The butterfat was incubated at 42°C. in open glass jars for four weeks and peroxide values determined at intervals. The results are given in Table 4.

TABLE 4.—ETHYL GALLATE AND KAMALA EXTRACT AS ANTI-OXIDANTS IN BUTTERFAT AT 42°C. INITIAL PEROXIDE VALUE OF CONTROL 0·08.

Treatment.	Lea Peroxide Values After—						
	3 days.	6 days.	9 days.	13 days.	16 days.	20 days.	28 days.
	%	%	%	%	%	%	%
Control	0·25	0·28	0·39	0·46	0·53	0·76	1·24
With 0·02 per cent. ethyl gallate	0·17	0·18	0·21	0·19	0·23	0·22	0·20
With 0·02 per cent. Kamala extract	0·17	0·23	0·22	0·22	0·21	0·24	0·28
Butterfat with 0·25 p.p.m. added Cu.	0·71	0·99	1·46	1·93	2·10	2·57	3·37
0·25 p.p.m. Cu with 0·02 per cent. ethyl gallate	0·12	0·16	0·19	0·20	0·19	0·22	0·23
0·25 p.p.m. Cu with 0·02 per cent. Kamala extract	0·23	0·29	0·32	0·49	0·52	0·59	0·76

Both anti-oxidants appeared to exert about the same effect in pure butterfat but the superiority of ethyl gallate was marked in the presence of added copper. Accordingly, an experiment was arranged to test the effect of this compound on the keeping quality of tinned butter.

Experimental butter containing 0·02 per cent. ethyl gallate was made in 30-lb. lots from commercial, choicest quality, pasteurized cream and vacuum processed after manufacture. In one case the ethyl gallate was added in aqueous solution at the grain stage in the churn and in another it was dissolved in fresh butterfat, and added during vacuum processing. The butter was sealed in 1-lb. tins and again stored at room temperature, being placed in addition in a 30°C. incubator for a period of five days in each month. Grade scores and comments are given in Table 5. The butter was salted in the usual manner, the dry salt being added at the grain stage.

TABLE 5.—EFFECT OF ETHYL GALLATE ON KEEPING QUALITY OF TINNED BUTTER.

Sample.	Grade Scores and Comments.		
	Initial.	5 weeks.	10 weeks.
Control	High acid, slight, stale and metallic 92 + pts.	Very stale—88 pts.	Stale and tallowy —85 pts.
With ethyl gallate 0·02 per cent. added to serum	High acid, slight, stale and metallic 92 — pts.	Stale—90 pts. ..	Very stale—87 pts.
With ethyl gallate 0·02 per cent. in butterfat	High acid, slight, stale and metallic 92 – pts.	Stale—89 pts.	Very stale—86 pts.

The salt contents of these butters varied from 2·9 to 3·1 per cent. on a 16 per cent. moisture basis. This, combined with vacuum processing, was sufficient to restrain bacterial growth as judged by total counts and the absence of characteristic bacterial flavours. It will be observed that there was a fairly consistent one or two points difference in grade, in favour of the butters containing the anti-oxidant. After five weeks, this score difference was rather impressive, as it resulted in a clear-cut differentiation between first and second grade. The difference was not so favourable on further holding when the quality of the three butters dropped to a poor second grade. It is clear that the effect of the anti-oxidant was to retard and not to stop the progress of general deterioration. The absence of a definite tallowy flavour in the presence of ethyl gallate even after ten weeks is of interest. Peroxide values on the three butters also suggested that the anti-oxidant had a very definite effect in controlling fat oxidation. The value for the control after four weeks was 0·61, whilst that of each of the butters containing ethyl gallate was 0·11. It was apparent from the grade scores, however, that destructive changes occur which are not inhibited by the anti-oxidant and which are intensive enough to lower the quality to at least a poor second grade. The ethyl gallate had an incidental effect on the colour of the butter. When freshly made, the treated butters were more neutral in colour than the control. After storage the difference was marked, butters containing ethyl gallate being of a greyish tinge, comparing unfavourably with the natural yellow of the control. The gallic acid reacted apparently with traces of iron in the butter; black spotting due to the same cause was very pronounced on interior tinplate and butter surfaces.

6. Conclusions.

The results of these experiments are not conclusive in regard to the control of bacterial defects in tinned butter, but they emphasize several factors of importance in this respect. These are the effects of butter texture and salt concentration, the types of organisms present, and the period and conditions of storage. In butter of poor texture, especially when associated with re-working, salt concentrations as high as 3·5 per cent. may fail to inhibit the development of rancid or cheesy flavours. Even with a very fine texture such as that achieved by vacuum processing, salt concentrations of 2·9 to 3·1 per cent. have been found necessary to give satisfactory control. There is some evidence to show that in the absence of resistant types of organisms, lower salt percentages combined with vacuum processing are effective. Boric acid in concentrations of 0·12 to 0·25 per cent. was found to exert a definite preservative effect. its addition to butter being accompanied by a consistent reduction in bacterial counts and an improvement in keeping quality. In this case the proportion of salt varied between 2·5 and 3 per cent.; it is probable that reports indicating the absence of preservative effect with this compound are due to the use of lower salt concentrations or less severe conditions of storage.

The use of compounds such as boric acid and ethyl gallate in butter raises questions which lie outside the scope of this discussion. The essential conclusion derived from experiments in which they were used was that in spite of some improvement in keeping quality, deleterious

flavour changes still occurred. On the whole, it was obvious that no substantial alteration to the essentially perishable nature of the product had been brought about. In all cases deterioration was observed to be progressive and there was no suggestion at any stage that quality could be stabilized at a level of palatability which would enable tinned butter to be safely stored and handled under the same conditions as other tinned foods. This conclusion is reinforced by consideration of the comparatively low melting point of normal butter and the disastrous effects of excessive temperatures on its condition and acceptability.

7. Acknowledgments.

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Report on Systematic Work on Red Algae in Australia.

*By Valerie May, M.Sc.**

Summary.

A brief survey has been made of the history of algal taxonomic studies in Australia.

It has been demonstrated that useful economic studies are dependent on knowledge of the taxonomy of the group concerned. At present there is a lack of adequate knowledge of the algae.

The procedure for studying algae is outlined, and various criteria for classification are discussed. Of these criteria, microscopic anatomy is proving of great use, while colour and absolute size are discounted.

1. Introduction.

The term algae covers a large, heteromorphic group of plants at a relatively low level of evolution. Algae include plants of marine, fresh water, terrestrial, and soil habitats, and range from minute, unicellular organisms such as diatoms and desmids to large, frondose plants such as the kelp *Macrocystis*. The term seaweed is usually restricted to include only the littoral (mainly macrosopic) algae.

In Australia, most work on the taxonomy of the freshwater microflora has been done by Playfair; this is not included in the following discussion.

A group of aquatic angiosperms is often confused with the seaweeds; this group includes such plants as *Zostera, Ruppia, Posidonia,* and *Cymodea.*

Among the seaweeds themselves there are four main groups, known as the Cyanophyceae (blue-greens), Chlorophyceae (greens), Melanophyceae (browns), and Rhodophyceae (reds). The first of these is relatively poorly represented in the sea and as a consequence has received very limited attention from most workers.

2. Historical Survey.

Modern systematic botany uses as its starting point Linnæus' *Species Plantarum,* first published in 1753. Here, under the heading of algae, Linnæus recognized various non-algal organisms such as liverworts, while of the true algae he distinguished only three genera, viz., *Fucus, Ulva,* and *Conferva.* In 1768 Gmelin published the first work devoted entirely to Algae, but generally speaking there was very little attention paid to the group until about 1840. By 1846 such calcareous algae as *Corallina* and *Halimeda* were just being recognized as being plant forms (Harvey, *Phycologia Brittanica*) and the European botanists were beginning to investigate collections from far-off colonies and dependencies.

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During the latter half of the last century, work was necessarily of an extensive rather than intensive nature, and the sorting of overseas collections and publishing of results became of great importance for the algologists in Europe. Noted names in this connection are those of Hooker, Agardh, Kuetzing, Sonder, and Harvey.

Harvey himself visited Australia and became the leader of, and for many years almost the solitary worker on, algal taxonomy here. In fact his published works are still in many cases our main source of information. In 1847-9 he published his *Nereis Australis* and in 1858-63 his *Phycologia Australica*—both series being illustrated, and the latter work providing the foundation for subsequent Australian work. Naturally the collections on which the work was based were far from complete and, of those plants recorded, the details given are often inadequate for the critical comparisons necessary in taxonomic work. At times Harvey rescinded his earlier findings; thus he described one plant as *Erythroclonium charoides* Harvey, but later he called this same plant *Rhabdonia charoides* Harvey, stating " nor did I discover my error until, having made a cutting for the present Plate, I found that the axile filament which characterizes *Erythroclonium* was not present."

This rapid work characterizes the period. There were floras of many newly discovered regions available, many collections were being made, and there was a lack of detail in the description of the voluminous material.

About this time attempts were also made by others to add to the knowledge of Australian algal plants. Lists were prepared by Sonder (1880-1) for Australia, by Harvey and Hooker (1847) for Tasmania, by Bailey (1895, 1909) for Queensland and by Wilson (1892) and by Tisdale (1898) for Victoria, while in 1898-9 Bastow attempted illustrated keys to the tribes and genera of Australian brown and red seaweeds.

A publication of world scope was De Toni's excellent *Sylloge Algarum*, the first volume of which appeared in 1889, the last volume in 1924. This, written wholly in Latin, is a compilation of preceding work and is of particular value for records, locations, and references.

All this earlier work, however, provided information which is very incomplete and fragmentary. It is only during the present century that more critical and detailed work on algae has been attempted, and the workers are so few that the re-sorting of most early material is still to be done.

In the present era, Setchell and Gardiner, of the University of California, have taken a lead in systematic work, and the many publications by them and their students are of great value; while Kylin, in a series of papers in Lunds Universitets Arsskrift, has given us much excellent work of general application to algal problems. For Britain, in 1931, Newton's " Handbook of British Seaweeds," summed up the position of classification, and Papenfuss has dealt with some African plants.

In New Zealand, the late R. M. Laing for many years carried on taxonomic studies on the algae of New Zealand and Norfolk Island, and at the present time Lindauer and Moore are both actively engaged on studies of seaweeds there. The names best known for work on algae in Japan are those of Yendo, Yamada, and Okamura.

In Australia extensive collections were made by A. H. S. Lucas, who published in *Proc. Linn. Soc. N.S.W.* a series of Notes on Australian Algae, including descriptions of new species. It is to him that is owed the good condition of the algal collection in the National Herbarium, Sydney. In 1909-12 he listed all known Australian algae (by then 1,050 species of reds and browns as against Harvey's 719 and Sonder's 923). He also published work on State floras, Tasmanian in 1915 and 1928, and South Australian, Part 1 (greens and browns only) in 1936. It is greatly to be regretted that Lucas did not live to publish his Part 2 (reds) of this series. His manuscripts are preserved, however, and the work was so far advanced that his co-worker, Mrs. Perrin, aims to have it published shortly. Occasional additions to our knowledge of the flora have been made by overseas workers such as Reinbold (1897, 1899), A. and E. S. Gepp (1906), and Tilden and Fessendon (1931).

In 1938-9, the writer published keys to the green and brown algae of New South Wales, further increasing the number of species listed as occurring in that State. At present, the main study concerns the red algae, which comprise a far larger group.

3. Taxonomic and Economic Studies.

In Australia, interest has now been centred on the algae for economic reasons. These are based primarily on the needs of agar production, but the production of alginates, &c., may be of importance later.

For agar production it has been found that some species give satisfactory yields, while others, superficially similar, are not satisfactory. With standardized chemical tests, this variation may be due to :—

   (i) Specific and varietal differences, i.e., genetic differences.
   (ii) Physiological conditions (age and stage) or changes in external environment or season.

The present study aims at clarifying the position with regard to (i). Failure to distinguish between closely allied species or varieties would, for instance, render chemical and physiological experiments futile; results obtained in different experiments would not be comparable if different species were being used. Further, without the means of reference provided by the systematic determination of the plant in question, i.e., its scientific name, it is futile to convey accurate information regarding its properties.

For all biological work the first essential is a good general systematic background. This can be achieved only by first gaining a wide familiarity with the organisms concerned, for on this basis there can be built a series of monographs on individual genera. It is only then that one can feel that the foundation work of taxonomy is sufficiently sound to enable other branches of science to build upon it.

As an example of the errors exposed when a monograph is prepared by an expert, Setchell's 1914 paper on the *Scinaia* Assemblage might be cited. Before he undertook this examination, the assemblage was thought to consist of six species and three varieties of *Scinaia*. When this material was more thoroughly investigated it was found to consist of three genera, of which the genus *Scinaia* alone contained eleven species and two varieties.

Mistakes are, however, not always in the direction of recognizing too few species. The writer's work on *Ectocarpus* (1939b) showed the wide variation of a single species, formerly known under two names.

4. Criteria Used in Taxonomic Analyses.

Difficulties in the delineation of species make essential a careful consideration of those criteria which can be used for the separation of closely related forms, since the characters used in the past are often inadequate and sometimes unsatisfactory.

When dealing with land plants, reproductive structures provide some of the most generally useful criteria for the determination of species. In the algae reproductive structures provide the basis for the classification of the large divisions, but the presence of reproductive material in a collection is relatively rare, so that other criteria must often be used for specific determination. In fact, many species have been described from incomplete material pending the discovery of reproductive stages. Thus more emphasis is placed on vegetative characters, and certainly the diversity of form found among the algae does lend itself to a classification on this basis. This method actually works fairly satisfactorily, provided that the anatomy is known.

Macroscopic characters such as general habit, form, degree of flattening, texture, presence of "midribs," method of branching, &c., are of definite use in the identification of a specimen. It must be emphasized, however, that external appearance alone is not sufficient. Field determinations without more detailed knowledge of the plant inevitably lead to serious errors, and hasty work of this nature is most unsatisfactory.

Size as a character has been used by some algologists, though the writer's experience suggests this to be unjustifiable, as plant size varies very much. Cell size has been used also, but the writer's observations on the measurements of cells of a filamentous alga *Ectocarpus* (1938) showed that in this case there must be a difference of at least 37 per cent. in cell size before the length of a cell becomes significant for separating species. Similarly, a large number of other species handled vary greatly in both cell and plant size. From the present studies it would appear that proportional measurements or ratios are more likely to be useful than any absolute measurement, though here again evidence is not yet conclusive.

Colour is another character which has been used widely by some workers, but which appears to be very unreliable. *Gracilaria* can serve as an example; here the colour is red, pink, yellow, light green, or nearly

black-green in one and the same species, variations occurring sometimes in the one plant. Other species also vary, though not so markedly, but the variations already known make the use of the colour criterion of doubtful reliability.

Secundity and distichy are both reliable criteria in some cases (e.g., *Plocamium* and *Laurencia* respectively), but not in others.

It is being increasingly recognized that anatomical structure has definite value for separating groups of plants. Quite a few genera of algae can be separated simply on the characters shown in cross sections of the thallus. Thallus structure varies in the number, relation, and arrangement of the component cell units, also in the hollow or solid structure of the thallus. Other anatomical differences, such as length-breadth ratio of peripheral cells, &c., and cell detail of the apical region, are also of use.

As in all taxonomic work, different characters are of different relative use in any particular group, and assessment of the value of any character can only be made when a detailed knowledge of the group has been gained. In the genus *Gracilaria* for example the method of insertion of branches varies considerably, and at first one is inclined to think this could be used as a diagnostic feature. Acting on this assumption the writer at first kept forms of *Gracilaria* separate if they varied in this character, even if they were otherwise alike. However, further study, especially in the field, induced the discard of this division as being wholly artificial. Branches which are inserted quite differently have been found growing on the one plant. In *Gracilaria*, therefore, the method of branch insertion has proved an unreliable criterion. This does not invalidate its use in other genera; for example in *Plocamium* it has proved very satisfactory.

The variation in form shown by many algal species is confusing enough in itself, but diagnosis of species is complicated further by the fact that in many algae there is a plant phase in the life history where reproduction is only by asexual spores. This phase may or may not vary in vegetative characters from those of the plants in which only sexual reproduction occurs. This dual nature was a cause of much confusion to early workers, and it is only in the last hundred years that this difficulty has been realized at all. Only recently plants previously believed to be parasites have been found to be part of the life cycle of the " host " plant. Cultivation under laboratory conditions is the obvious way to solve problems of this sort, for under cultivation the complete life cycles may be observed. In fact, if artificial culture can be carried out more extensively, it is likely to prove of great value also in distinguishing those variations which are entirely due to environmental differences from those due to genetic factors. Culture work may for instance demonstrate a correlation between some such character as physiological stage or assimilation rate, and some of the apparently haphazard variations now known to exist. Some seaweeds are already known to vary in appearance with depth and exposure to surf, &c. The growing of red algae under controlled conditions is, however, a relatively unexploited field.

5. Procedure of Study.

Taxonomic work must be preceded by adequate field work and exten-
sive collecting. With each collection, notes are made of the location,
date, and any information available as to the habitat. As wide a range
as possible of the material available is collected and, all possible
information from the fresh material having been obtained, the latter is
sorted macroscopically into its obviously different species and prepared
for later more detailed examination. Two methods of preservation are
used: (i) pickling in bottles, and (ii) the preparing of dried herbarium
mounts. The former method has the advantage of keeping the plant
material as near as possible to its original form, but the disadvantage is
that it is less easily stored as a permanent record. One is now ready
for the microscopic examination and critical classification of the plant
material.

In the present incomplete state of our knowledge, the method of
tackling " unknowns " which has been found most satisfactory by the
writer is to treat all minutely different forms, whether from field collec-
tions or herbaria, as distinct and to group these forms into " species "
only when and if adequate material and field work have erased the
dividing barrier.

The individual " species " thus separated can then be compared with
named specimens in available collections. In all cases, available litera-
ture must be examined to compare the unknown with the original type
description and specimen, especially as the specimens in existing herbaria
are by no means always correctly identified. This basic work must
be done sooner or later if Australian algology is to attain status. Until
this work is completed identifications must necessarily be provisional.

In the attempt to identify collected " species " with names already in
use all available records were collected, from literature and herbaria,
of the species of red seaweeds occurring in New South Wales and Queens-
land waters, the only areas in which the writer has yet worked.

In view of the inadequacy of our existing knowledge of the algae it
is only to be expected that, in the course of extensive collecting, new
species and varieties will be found, and that new records of species
known in other parts of the world will be made from time to time.
Naturally, many of the samples received are repetitions of a few
common " species " and as such are becoming routine identifications.
These duplications are of value in the compilation of seasonal and
distributional information, which is being accumulated as the result of
this work.

The genus *Gracilaria*, on which interest is mainly focused at present,
resembles, in vegetative features, certain forms of *Hypnea*. When one
compares relatively mature plants, these genera can be distinguished on
vegetative characters, as can the various species of *Gracilaria* itself.
In very juvenile or incomplete portions of plants, however, the distin-
guishing characters may be missing. This shows the necessity for
adequate collecting by competent collectors. It is obvious that the more
familiar a collector is with the group concerned the more capable he is
of making a suitable collection.

6. Avenues for Further Studies.

Japan has progressed farthest in the economic utilization of the
" pastures of the sea." Here the ecology has been studied from the
economic point of view, and the important weeds are cultivated in much
the same way as oysters are in this country—the right substrate is
planted at the right time and in some cases the cultures may be trans-
ferred to environments more favourable to bulk growth.

In the past the controlling factors in the biology of an alga have
been considered to be geological formation, temperature, depth, salinity,
illumination, water movement, aeration, physical substrate, topography,
and the meteorological history of the area. It is only by determining
the relative importance of these various factors in any species concerned
that we will be able to determine areas in which that species could be
made to flourish.

Marine ecology, dependent on a sound algal taxonomy, is a study
which may well merit attention at a later date. Attempts were made by
Hedley (1915) and Pope (1943) to describe the relation between the
algae and marine animals of the same zone, but much yet remains to be
done. Great advances can be expected in work of this kind.

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Solubility of Nicotine in Aqueous Salt Solutions.

By J. S. Fitzgerald, M.Sc., Ph.D.

1. Introduction.

During investigations of the commercial methods for extracting nicotine from tobacco waste, it was seen that very little use is made of the peculiar solubility relations of nicotine and water. This system has been investigated by Hudson (1904) and by Tsakalotos (1909), and is the classical example of a closed solubility curve, having both upper and lower critical solution temperatures. Maximum separation occurs about 95°C. when the two layers contain 6·5 per cent. and 82 per cent. of nicotine respectively. Timmermans (1907) has shown that in binary systems mutual solubility is decreased by the addition of substances soluble in only one of the components, but this principle of salting-out has not been applied to the concentration of nicotine solutions.

Two fairly recent patents (McConnell 1930; Klose 1938) claim what amounts to such a salting-out process, but neither gives detailed information regarding the equilibria. The former aims at preparing free nicotine from solutions of its salts, preferably by addition of hydroxides which give insoluble salts with the acid radicle. It is not directed towards concentration of the nicotine in the absence of acid. The German patent is essentially the same, but specifies a large excess of alkali carbonate as the means for liberating the free alkaloid.

The effect of sodium hydroxide or sulphate on the critical solution temperature with a small range of nicotine concentrations near the lower critical solution point has been studied by Dubrisay (1922). He showed that the lower critical solution temperature was depressed by the added salts. Cuvelier (1936) studied the influence of the several ammonium salts on the nicotine-water system, and his data cover the lower nicotine concentrations which must be considered in assessing the efficiency of a salting-out process. His graphs indicate that the less concentrated of the two layers obtained by heating the ternary mixture has decidedly less than the 6·5 per cent. nicotine obtained from water and nicotine alone. However, since nicotine, though a weaker base than ammonia, is not of a very different order of strength, it can be shown that the equilibria established between ionized and unionized forms of ammonia and nicotine will tend to reduce the salting-out. Owing to the great difference in basicity between caustic alkalis and nicotine, alkali salts will not have this complicating effect, and the differences found between ammonium and caustic alkali salts in the present work support this conclusion.

Measurements of interfacial tension and critical solution temperature by Ssementschenko *et al.* (1934) confirmed the theoretical requirements that substances that raise the interfacial tension cause the region of partial miscibility to increase. Of the salts used, the following is the order of increasing effectiveness in depressing the lower

critical solution temperature of aqueous nicotine solutions: lithium bromide, sodium bromide, sodium chloride, sodium fluoride, magnesium sulphate (sodium iodide raises the C.S.T.).

It is of interest to note that the opposite effect has been demonstrated by Leone (1926) who added acetone, a substance miscible with both components, and found the region of heterogencity decreased as the acetone concentration increased.

2. Experimental.

(i) *Materials.*

The salts used were all of B.D.H., A.R. quality. The nicotine was B.D.H. pure, redistilled twice before use, the fractions taken in one instance (for example) boiling at 127°–129°C./25mm. and 125–126°C./22mm.

(ii) *Methods.*

For the determination of the lower clouding temperatures, the synthetic method was used. In tests on the binary system water-nicotine, a stock solution was made up by weight, a portion removed and sealed in a tube, then further samples prepared by successive dilutions of the stock with water until a trial sample showed no turbidity on heating almost to boiling. With the ternary mixtures the same process was used, but dilution was carried out using a salt solution of the same concentration as in the ternary mixture. For some higher nicotine concentrations mixtures were weighed into the individual tubes. For both binary and ternany mixtures the sealed tubes were heated in a water bath where they could be observed under rising and falling temperature, being tilted continually to ensure good mixing. Several readings were taken for each observation. The complete range of concentrations was not always covered in one series of dilutions.

Determinations were also made by the synthetic method by adding relatively concentrated salt solution to nicotine solutions and noting the clouding temperature after each addition. The results cannot be directly graphed as nicotine and salt concentrations are varied simultaneously, but by interpolation from the three graphs of the data taken two variables at a time, the corresponding figures at either constant salt concentration or at constant temperature may be found.

The solubility of the salt in concentrated nicotine solutions, and the solubility of nicotine in the more concentrated salt solutions, set limits to the determinations which could be made by the synthetic or plethostatic method, and so nicotine in the more concentrated layer was determined by analysis of mixtures separated at constant temperature. It may be mentioned here that Hudson (*loc. cit.*) found that the relative densities of the two aqueous nicotine layers were reversed above 90°; owing to the increased density of the dilute layer due to the added salt there is no similar reversal in the ternary mixtures. Evaporation of some samples of the concentrated nicotine layer showed no residue, indicating that all the salt is contained in the more dilute

layer, but when solutions were separated at lower temperatures, appreciable amounts of salt were found in the nicotine-rich layer. Only a few determinations were made, and these showed that the concentration of salt in aqueous nicotine decreased rapidly with increasing nicotine concentration.

(iii) *Analytical Methods.*

The usual method for precipitating barium sulphate was used for estimating sulphate, after it had been shown that the nicotine did not interfere in the dilutions used. In the analyses of the concentrated nicotine layer small samples only were used, and the results are not very accurate at the lower sulphate concentrations, as is indicated in Table 8.

For determinations of nicotine, a modified picrate method was used. Nicotine dipicrate has been considered by other workers to be less desirable than the silico-tungstate for quantitative analysis because of the greater solubility of the picrate in the absence of excess picric acid, and the difficulty of washing the precipitate free of picric acid when excess is used. By using a large excess of picric acid good results were obtained except when very small amounts of nicotine were used, and the precipitate was freed from picric acid by the use of ether as a wash liquid following washing with the minimum quantity of half-saturated picric acid.

3. Results.

Tables 1 to 5 show the clouding temperatures observed at different nicotine concentrations, the figures in brackets being those interpolated from graphs of the results obtained by the analytical method, though for the binary mixtures the experimental analytical figures are given. The clouding temperatures found when both salt and nicotine concentrations were varied are shown in Table 6, and the interpolations for constant temperatures are summarized in Table 7 having been read from graphs of the salt-temperature and nicotine-temperature data. Results of the analytical determinations are given in Table 8. In this table, figures interpolated from information obtained by the synthetic method are given in brackets.

TABLE 1.—NICOTINE—WATER.

Nicotine.		Clouding Temperature.	Nicotine.		Clouding Temperature.
%		°C.	%		°C.
(79)	.. ..	(97)	28·07	.. ..	62·4
(76)	.. ..	(84)	26·7	.. ..	61·5
(71)	.. ..	(74)	24·99	.. ..	62·3
(64·0)	.. ..	(70)	20·12	.. ..	63·8
58·2	.. ..	63·8	15·88	.. ..	66·0
48·85	.. ..	62·2	13·12	.. ..	69·0
44·4	.. ..	61·4	10·22	.. ..	73·0
40·19	.. ..	61·1	8·32	.. ..	79·5
37·6	.. ..	62·2	6·76	.. ..	88·5
32·84	.. ..	60·8	5·43	.. ..	—
29·3	.. ..	61·5			

TABLE 2.—NICOTINE—WATER—SODIUM SULPHATE.

0·29M. Na₂SO₄. (3·93 %).		0·57M. Na₂SO₄. (7·5 %).		1·07M. Na₂SO₄. (13·2 %).	
Nicotine.	Clouding Temperature.	Nicotine.	Clouding Temperature.	Nicotine.	Clouding Temperature.
%	°C.	%	°C.	%	°C.
(15 0)	(30)	6 44	24 3	1·52	28 4
13·78	32·2	(5·25)	(30)	1·22	42·5
(10·5)	(40)	5·18	31·6	1·0	58·3
10·0	42·9	4·13	40·1	0·9	68·2
8·0	49·5	(4·0)	(40)	0·8	86·5
(7·5)	(50)	(3·25)	(50)	0·7	—
6·0	57·2	3·15	51·9		
(5·25)	(60)	(2·75)	(60)		
(4·25)	(70)	2·52	63·9		
(4·25)	73·9	(2·25)	(70)		
(3·5)	(80)	2·09	80·1		
3·23	98·3	(2·0)	(80)		
2·99	—	1·79	102·0		
		1·54	—		

TABLE 3.—NICOTINE—WATER—POTASSIUM SULPHATE.

0·029M. K₂SO₄. (0·5 %).		0·058M. K₂SO₄. (1·0 %).		0·146M. K₂SO₄. (2.47 %).		0·29M. K₂SO₄. (4·82 %).	
Nicotine.	Clouding Temperature.	Nicotine.	Clouding Temperature.	Nicotine.	Clouding Temperature.	Nicotine.	Clouding Temperature.
%	°C.	%	°C.	%	°C.	%	°C.
54 41	(Salt pptd.)	46 3	(Salt pptd.)	23 8	39 1	9 68	41 3
52·35	37·5	43·58	33·8	15·34	47·7	7 13	49·7
50 27	41·9	42 46	36 9	10 78	54 7	5 25	60 6
45 39	49·8	39 82	42 2	8 37	60 8	4 31	69 1
40·97	53·2	37 24	46 7	6 19	71·0	3 52	81 1
35·36	55·6	28·98	50·7	5·04	81·2	3 09	95·0
30 05	56·8	17 77	56 6	4 11	97 2	2 81	108 6
18·78	60·5	12·65	61·7	3·37	—	2·54	—
13·15	65·4	9·18	69·1				
9·39	73·6	7·35	77·2				
7·38	81·6	6·67	81·1				
6 67	87·0	6·08	85·2				
5 95	93·6	5 53	91 8				
5·25	107·0	5·02	101·0				
4·56	—	4·55	—				

TABLE 4.—NICOTINE—WATER—AMMONIUM SULPHATE.

0·20M. (NH₄)₂SO₄. (2·5 %).		0·40M. (NH₄)₂SO₄. (5·0 %).	
Nicotine.	Clouding Temperature.	Nicotine.	Clouding Temperature
%	°C.	%	°C.
40·39	23·5	24·57	24·9
37·48	32·6	19·78	32·3
35·11	36·5	14·98	39·2
32·15	39·5	9·8	51·3
28·8	42·4	7·96	59·8
24·13	45·8	7·17	65·9
19·78	49·1	6·47	74·8
14·84	53·9	5·82	85·7
8·9	68·8	5·49	97·0
7·91	74·4		
6·3	92·0		
5·4	—		

TABLE 5.—NICOTINE—WATER—SODIUM CARBONATE.

0·93M. Na₂CO₃. (8·92 %.)

	Nicotine.	Clouding Temperature.
	%	°C.
1·59	..	42·7
1·35	..	51·0
1·12	..	65·0
0·94	..	87·0
0·88	..	108·0
0·79	..	—

TABLE 6.—NICOTINE—WATER—SODIUM SULPHATE.

Experiment.	Nicotine.	Na₂SO₄.	Temperature.	Experiment.	Nicotine.	Na₂SO₄.	Temperature.
	%	%	°C.		%	%	°C.
A	21·1	0·9	52·5	D	30	0·94	49
	20·0	1·3	49·0		28·4	1·28	43·5
	19·6	1·5	47·0		27·2	1·64	39·5
	18·7	2·0	43·5		25·9	1·95	36·25
	17·6	2·4	41·0		24·7	2·23	33·5
	17·5	2·6	40·0		23·7	2·50	30·7
					22·7	2·74	28·5
B	26·7	0	61·5	E	37·6	0	62·2
	24·2	0·89	51·6		35·7	0·48	53·3
	22·1	1·62	44·7		34·0	0·92	46·3
	20·4	2·24	39·7		32·3	1·30	40·5
	18·9	2·76	36·3		30·8	1·66	35·5
	17·6	3·22	33·3		29·4	1·98	31·4
	16·4	3·62	31·5		28·2	2·28	27·8
	15·5	3·96	29·6		27·0	2·55	24·6
C	29·3	0	61·5	F	44·4	0	61·4
	27·8	0·44	56		42·1	0·44	48·9
	26·5	0·83	51·3		40·0	0·85	37·8
	25·4	1·19	47·8		38·2	1·22	24·9
	24·2	1·52	44·2				
	23·3	1·83	41·7				
	22·5	2·11	39·4				
	21·6	2·38	37·3				
	20·8	2·61	35·4				
	20·1	2·84	33·9	G	58·2	0	63·8
	19·6	3·04	32·4		55·3	0·18	51·4
	18·8	3·2	31·0		51·4	0·43	29
	18·1	3·42	29·9		50·5	0·5	27·9

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TABLE 7.—NICOTINE—WATER—SODIUM SULPHATE.
(Interpolated from graphs of Table 6.)

30°C.		40°C.		50°C.		60°C.	
Na₂SO₄.	Nicotine.	Na₂SO₄.	Nicotine.	Na₂SO₄.	Nicotine.	Na₂SO₄.	Nicotine.
%	%	%	%	%	%	%	%
3·9	15·75	2.6	17·5	1·15	20·5		26·25
3·4	18·25	2.2	20·5	1·05	23·75	var.	29
2·6	23·25	2·05	22·75	0·95	26	0·15	32·5
2·1	29	1·6	27·25	0·9	30·25	to	37·5
1·1	39	1·3	32	0·7	35·0	0·05	44
0·5	51	0·75	40·5	0·4	42·5		57·5
		0·4	53	0·25	55		

TABLE 8.—NICOTINE—WATER—SODIUM SULPHATE.
(Analytical Method.)

Temp.	Dilute Layer.		Conc. Layer.		Temp.	Dilute Layer.		Conc. Layer.
	Na₂SO₄.	Nicotine.	Na₂SO₄.	Nicotine.		Na₂SO₄.	Nicotine.	Nicotine.
	%	%	%	%		%	%	%
30°C.	3·9	15·2	1·7	39	60°C.	0·6	..	53·3
	(3·93)	(14·25)	..	..		1·0	17·0	57·5
	4·8	11·1	1·2	40		1·2	14·75	61·6
	(7·5)	(5·5)	..	..		2·2	8·7	62·8
	8·3	3·3	0·6	52·5		3·25	6·2	64·7
	9·4	2·1	0·5	54		(3·93)	(5·7)	..
	10·9	2·5	0·55	56		4·6	4·5	66·85
	11·7	1·9	..	57·6		7·1	2·9	71·8
	(13·2)	(1·5)	..	..		(7·5)	(2·7)	..
	16·2	0·5	..	66·2		8·95	2·0	73·0
						(13·2)	(1·0)	..
40°C.	2·9	14·2	0·7	41·5		14·4	0·8	79·4
	(3·93)	(11·0)	..	..				
	4·0	10·0	0·4	48	70°C.	0·2	..	63·9
	4·3	9·6	..	50·5		1·1	9·5	65·1
	6·9	4·65	..	..		2·3	6·2	69·0
	(7·5)	(4·05)	..	..		(3·93)	(4·6)	..
	7·85	3·6	..	58·2		5·0	3·4	74·5
	10·85	2·0	..	61·4		(7·5)	(2·3)	..
	12·1	1·9	0·2	..		9·7	1·5	78·3
	(13·2)	(1·2)	..	..		(13·2)	(0·9)	..
	13·7	0·95	..	66·2		14·0	0·75	79·7
	15·1	0·85	0·15	67				
50°C.	1·5	21·1	0·4	43				
	2·35	11·75	..	52·3				
	2·85	11·7	..	..				
	3·2	8·55	..	56	80°C.	1·95	5·4	76·8
	(3·93)	(7·7)	..	..		(3·93)	(3·9)	..
	5·2	5·3	..	60·7		4·9	3·0	80·1
	7·2	3·6	..	63·9		(7·5)	(2·0)	..
	(7·5)	(3·25)	..	..		9·4	1·3	..
	10·4	1·95	..	68·2		9·85	0·8	84·7
	(13·2)	(1·1)	..	..		(13·2)	(0·8)	..
	14·3	0·9	..	73·0		13·9	0·75	83·8

TABLE 9.—DISTRIBUTION OF NICOTINE BETWEEN WATER AND
KEROSENE.

Aq. Layer.				Original Concentration Nicotine in Aq. Layer.	Concentration in Water/Concentration in Kerosene at Equilibrium.
				g./100 ml.	
Water	..	..	..	0·94	0·775
Water	..	..	..	1·82	0·785
Water	..	..	..	4·35	0·978
Water	..	..	..	8·83	1·07
1· per cent. Na_2SO_4 ..		..	..	1·95	0·60
5 per cent. Na_2SO_4 ..		..	..	1·82	0·38
10 per cent. Na_2SO_4		..	..	1·82	0·23
1 per cent. Na_3CO_3 ..		..	..	0·89	0·59
2 per cent. Na_3CO_3 ..		..	..	0·88	0·44
3 per cent. Na_3CO_3 ..		..	..	0·90	0·41
4 per cent. Na_3CO_3 ..		..	..	0·85	0·35
8 per cent. Na_3CO_3 ..		..	..	0·85	0·16

4. Discussion.

The correspondence between the results given in this paper and
those previously reported for the nicotine-water system is shown in
Fig. 2 and 3, and is quite satisfactory.

Fig. 1 shows a number of comparisons. It is evident that there
is little difference between the effects of equimolal concentrations of
sodium carbonate, sodium sulphate, and potassium sulphate. Dubrisay's
figures for 0·1N sodium hydroxide and 0·1N sodium sulphate, whilst
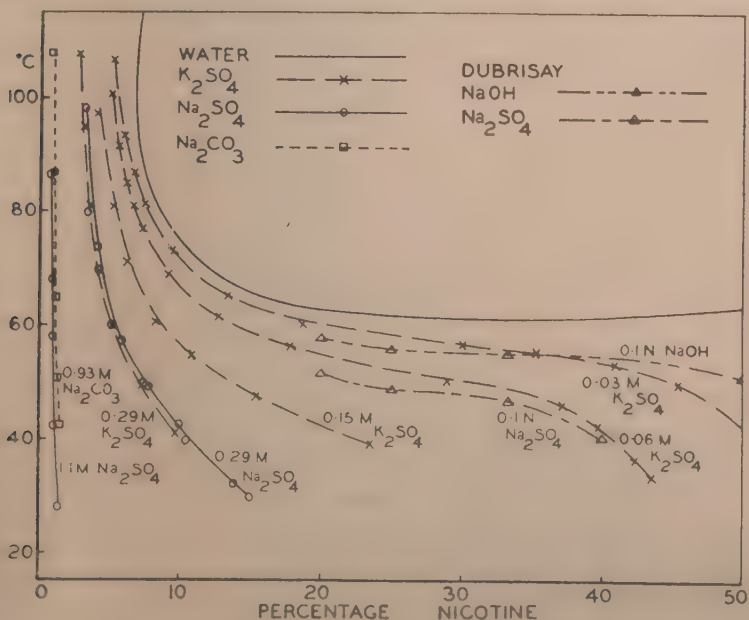
FIG. 1.—Solubility of nicotine in various salt solutions.

not directly comparable with the present results show the greater salting-out effect of the sulphate. The form of the potassium sulphate curves shows the decreased effect of temperature with increasing salt concentration.

Ammonium sulphate curves are shown in Fig. 2, where the present results are shown with curves interpolated from Cuvelier's graphs (he gives no figures) at equal molal concentrations, assuming his g.mols./100g. refers to solvent weight. The present results indicate concentrations of nicotine $0 \cdot 5$ to $1 \cdot 0$ per cent. lower than Cuvelier's at the higher temperatures, but even then comparison with the graphs for other salts shows ammonium sulphate is much less effective, particularly at the higher temperatures. The curve for $0 \cdot 29$ molal sodium sulphate has been drawn for comparison in the absence of figures for either of the molalities used with the ammonium salt, this concentration being half way between those used for the ammonium salt. The difference at the higher temperatures is very marked.
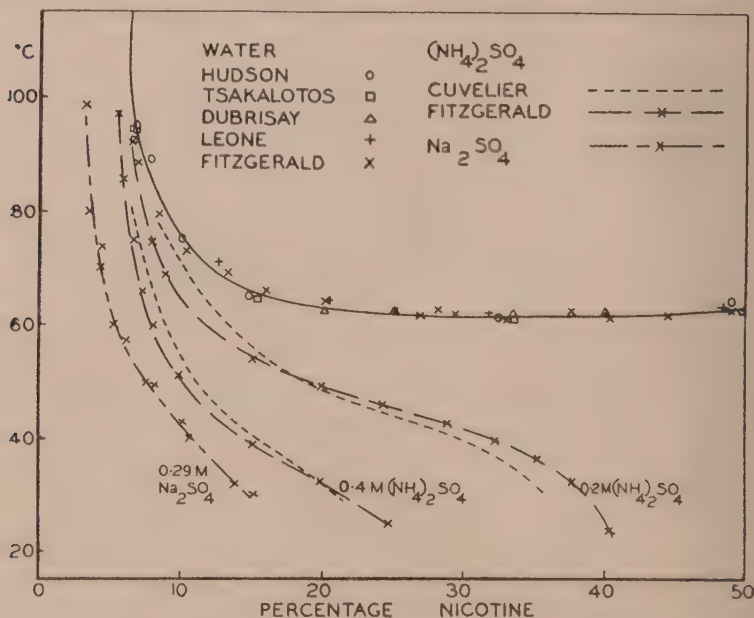
Fig. 2.—Solubility of nicotine in ammonium sulphate solutions.

The results of both synthetic and analytical work are shown in Fig. 3, where the nicotine-temperature relationship is graphed for various concentrations of sodium sulphate. Values found experimentally are indicated for the binary system, showing the agreement between the different workers, and also for the clouding temperatures from the present results for the ternary mixtures. The curves of the conjugate points corresponding to the nicotine-rich components separated at these clouding temperatures are also shown in Fig. 3.; they were obtained from the nicotine-salt graphs of the analytical results (given in Table 8) by interpolating at the appropriate salt concentrations. The curves indicate the nicotine concentration in the upper layer when the stated salt concentration is present in the lower layer.
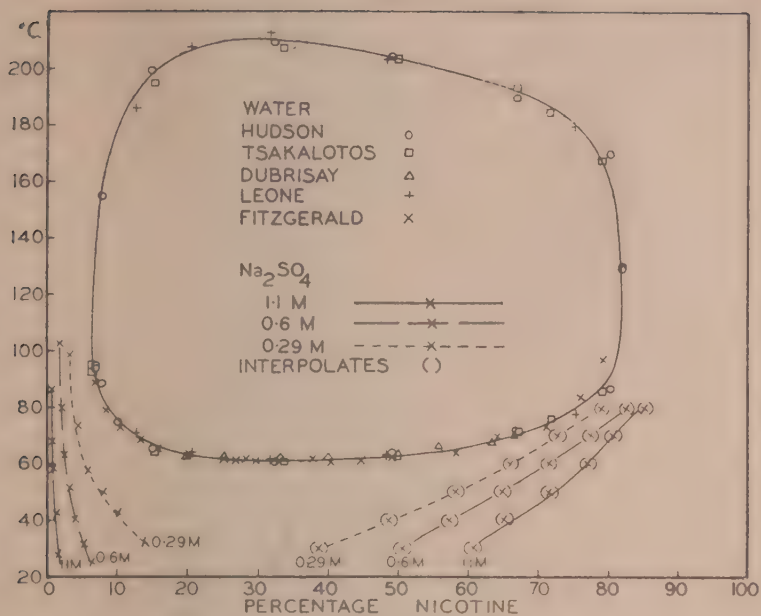
Fig. 3.—Solubility of nicotine in water and sodium sulphate solutions, showing conjugate curves.

Analytical results obtained at 30°C. are shown in Fig. 4 which is a distorted fraction of a triangular graph. The tie lines of conjugate points are shown. Lone points obtained by the synthetic method are
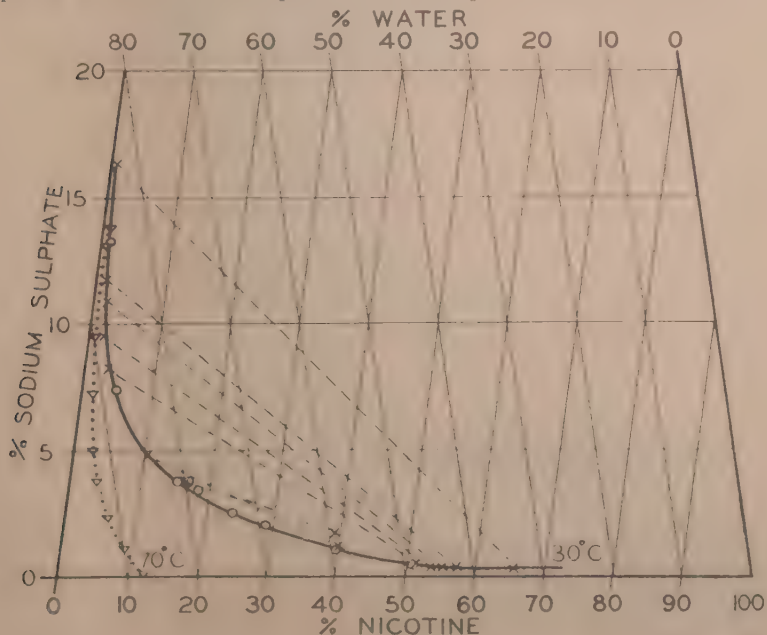

Fig. 4.—Solubility of nicotine in sodium sulphate solutions at 30° and 70°C., indicating conjugate points.

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also shown, as they indicate the whole curve more completely. The curve for results at 70 C. is also given, but as the salt concentrations in the upper layer are virtually zero in all cases, these have not been shown.

5. Application.

It is not the purpose of this paper to discuss the application of the results to any process of concentrating nicotine but it will be obvious that the solubility of nicotine may be very greatly reduced and a considerable amount salted-out. Where the primary separation is by a process such as steam-distillation, the dilute residue may be stripped, the salt solution aiding the liberation of the nicotine from the plant material. Methods for avoiding the loss of all salt after each separation, and for making a continuous process, will depend largely on the complete process of which the salting-out will be but one step.

The rapid drop in solubility of nicotine even at moderately low temperatures, points to the addition of salt as an aid to solvent extraction. From the literature of solvent extraction of nicotine, it is obvious that cheap solvents such as kerosene are poor extractants. The figures given in Table 9 show that the effect of added salt on the partition of nicotine between water and kerosene at about 20 C. is to drive the nicotine into the kerosene.

6. Acknowledgments.

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NOTES.

Building Materials Research.

With a view to conducting investigations into the development, manufacture, properties, and uses of building materials, the Council recently appointed Mr. Ian Langlands, M.Mech.E., B.E.E., as Officer in-Charge, Building Materials Research.

His first duties will be to make a survey of building industry problems to ascertain those which are most urgent and those whose solution can be sought with existing facilities. For the present, no Building Materials Research Laboratory will be established but experimental work will be carried out in existing laboratories of the Council, especially those of the Divisions of Forest Products, Industrial Chemistry, Aeronautics, and Soils and the National Standards Laboratory.

It is proposed to set up, as soon as possible, an information and intelligence service, the functions of which will be to survey and classify for easy reference the great volume of existing knowledge (both overseas and local) regarding building materials and their uses, to keep abreast with new knowledge as it becomes available, to digest the information and issue it in a form suitable for use by the building industry, and to deal with inquiries.

The work of the Council will be complementary to that of the Commonwealth Experimental Building Station of the Department of Post-War Reconstruction which will deal with the design and construction of buildings and their fittings. The work of these organizations is closely related and intimate co-operation and liaison will be maintained between them.

Mr. Langlands joined the Division of Forest Products in 1929, and since 1931 he has been Officer-in-Charge of the Timber Mechanics Section. For the present he will be located at the Division of Forest Products, 69 Yarra Bank-road, South Melbourne.

Appointment of Assistant Executive Officer.

Dr. F. W. G. White, formerly Professor of Physics in Canterbury University College of the University of New Zealand, has been appointed to the recently-created position of Assistant Executive Officer at the Council's Head Office.

For the last four years, Dr. White has been Chief of the Council's Division of Radiophysics, Sydney, having been lent by the University of New Zealand for that purpose. He obtained the Ph.D. degree of the University of Cambridge in 1931 and, after that, was Lecturer in Physics at King's College, University of London, until 1936, in which year he took up his professorial Chair at Christchurch. Early in 1941 the University of New Zealand was asked by the Australian Government to permit him to come to Australia to take charge of the Council's Radiophysics Laboratory which was brought

into being at the beginning of the war to carry out special research in radar (radio-location) for the Australian fighting forces. Throughout his career, Dr. White has carried out various researches concerning the physics of the upper atmosphere and radar.

Conference with United Graziers' Association of Queensland.

Executive Committee and other officers of the Council, including the Chiefs of the Divisions of Animal Health and Production, Biochemistry and General Nutrition, Economic Entomology, and Plant Industry, together with representatives of the Queensland State Committee, met representatives of the United Graziers' Association of Queensland, the Queensland Department of Agriculture and Stock, the Bureau investigating matters of Land and Water Resources of Queensland, and the University of Queensland, in Brisbane on Friday, December 1, 1944, to discuss various matters of interest to graziers in Queensland.

The main problems under discussion were the establishment of a pasture research laboratory at St. Lucia; the establishment of a scientific investigational committee to be called provisionally the Animal Industry Research Committee; the establishment of a drought feeding committee; and the need for research in problems of special interest to Queensland, such as noxious weeds, soils, the selection of edible shrubs and trees, the deterioration of natural grasses in the western or drier areas, sheep branding fluids, problems of animal physiology and nutrition, and the improvement of dehydrated meat.

Regional Research Centre in the Riverina.

Consideration has been given to the problems confronting the agricultural and pastoral industries in the large and important area of the Riverina, and to the means whereby effective scientific assistance can be given to these industries. Within this territory, irrigation schemes, which ultimately will involve the partial irrigation of several millions of acres of land hitherto used almost exclusively for pastoral purposes, are projected and are in process of development.

The Divisions of Soils, Plant Industry, and Animal Health and Production have given consideration to the problems associated with the use of irrigation water on partially-irrigated holdings (in the Riverina each block holder will be allowed sufficient water for one-tenth of his holding), and to the facilities that are necessary for the solution of these problems. The more urgent problems may be summarized as follows :—

  (a) Soils investigations: Detailed soil surveys to supplement and expand the existing and contemplated reconnaissance soil surveys in the New South Wales and Victorian irrigation areas; problems of water penetration; movement of deleterious salts; soil structure in relation to water usage and drainage on the major soil types.

(*b*) Plant investigations: Methods of land preparation for irrigated pastures; species and mixture studies; forage crops; fertilizer requirements, including trace elements; the management of irrigated and non-irrigated pastures severally and in conjunction; the relation of water usage to pasture production.

(*c*) Animal investigations: The provision of a balanced food supply; the production and storage of drought fodders; the efficient use by the animal of the products of irrigated agriculture.

(*d*) Irrigation investigations: The study of the best irrigation methods in respect to soil type, and kind of crop and pasture; maintenance of soil structure and fertility.

F. S. Falkiner and Sons Pty. Ltd. have given the Council an area of 1,550 acres of land near Deniliquin for the establishment of a field station to investigate these problems. The Council hopes to secure an additional adjoining area so that the station may be representative of the typical grey and red soils of the Riverina. In addition to this field station, the Council intends to establish a laboratory in Deniliquin to provide facilities for detailed soil and plant studies.

The research centre will work in close collaboration with the N.S.W. Department of Agriculture, and the Water Conservation and Irrigation Commission of N.S.W.

Review.

"Advances in Grassland Husbandry and Fodder Production."

This Bulletin is a symposium containing the type of material formerly published in *Herbage Reviews* and more recently in the Supplement to *Herbage Abstracts*. It is proposed to continue to publish collections of miscellaneous material in the form of Bulletins appearing at irregular intervals until such time as it is practicable to revive *Herbage Reviews*. *Herbage Abstracts* will continue to be issued in six parts per annum, of which five will be abstract issues and the sixth will contain the annual indexes.

The present Bulletin contains fourteen items on many aspects of the problems of grassland management and fodder production in different parts of the world. The items of greatest general interest will be the article by D. Meredith, of African Explosives and Industries Ltd., giving his views on the future of pasture management in South Africa, the review of the pasture aspects of the survey of the dairy industry in New Zealand made by W. M. Hamilton (in which he discusses the possible future expansion of the industry and its relation to production and requirements in other parts of the world), and the

statement of the crisis in the production and supply of seeds of herbage plants that has been experienced in the U.S.A., which has recently been the concern of a Committee appointed under the chairmanship of C. R. Enlow.

Amongst other items is a review of a recent report from Canada on prairie farm rehabilitation, in which a programme of regrassing ploughed-up and now abandoned land and the formation of " community pastures " of 15,000 to 20,000 acres in size are important aspects. A full translation of a Russian article on the irrigation of lucerne is included to indicate current views, practices, and recommendations regarding this type of fodder production in that country. Trials of herbage plants in New Zealand are described, and information on fodder trees in Madras is tabulated. Vegetation and soil conservation in Brazil and Argentina are reviewed.

Recent Publications of the Council.

Since the last issue of this *Journal,* the following publications of the Council have been issued :—

Bulletin No. 180.—" Studies on Deglutition in Sheep. 1.—Observations on the Course Taken by Liquids through the Stomach of the Sheep at Various Ages from Birth to Maturity," by R. H. Watson, D.Agr.Sc. " 2.—Observations on the Influence of Copper Salts on the Course Taken by Liquids into the Stomach of the Sheep," by R. H. Watson, D.Agr.Sc., and I. G. Jarrett, B.Sc.

The stomach of a sheep has four compartments—the paunch or rumen, the honeycomb or reticulum, the bible or omasum, and the rennet or abomasum; solid food normally passes through each in turn, but in the young lamb milk may by-pass the first three and go straight to the abomasum. Drenches given to sheep may pass to any or all four of the compartments.

As most drugs given in dosing sheep for worms are more effective when they pass straight to the abomasum and are not diluted in the other compartments, many attempts have been made to discover what governs the course followed by foods and liquids taken by the sheep, but much confusion on the subject remained. Certain substances, in particular copper sulphate, had been shown to affect the course taken, but the mechanism was not fully understood. The work reported in the present Bulletin was undertaken to obtain more information on these points in an attempt to clarify the existing confusion.

Suspensions of barium sulphate in milk or water were given to sheep and the course taken by the barium was observed by means of X-rays. It was found that when the sheep drank the suspension in response to an appetite for milk it usually passed direct to the abomasum, but when they drank merely to quench thirst it almost all went to the rumen and reticulum. When the suspension was administered as a drench, the course taken varied in different sheep, but it passed more frequently to the abomasum; when it was given shortly

after a solution of copper sulphate or other copper salt. It was known that copper sulphate had this effect, but the present work has shown that it is possessed by other copper salts also, and has provided information as to the conditions under which drenches may be expected to pass to the abomasum most frequently. The information gained is, too, of importance in the study of digestive processes in the sheep.

Bulletin No. 181.—" Sheep Blowfly Investigations. The Attractiveness of Sheep for *Lucilia cuprina,*" by I. M. Mackerras, M.B., Ch.M., B.Sc., and M. J. Mackerras, M.B., M.Sc.

This Bulletin describes investigations into the reasons why blowflies are attracted to living sheep. Many more sheep are struck in Australia by *Lucilia cuprina* than by any other blowfly. This is because *Lucilia cuprina* lays eggs on sheep far more rapidly than other species. Although other flies seldom lay eggs on living sheep, maggots of special species will develop as well as those of *Lucilia cuprina* if they are placed on sheep.

Two distinct sets of conditions are necessary for the production of strikes—those which attract the fly and stimulate it to lay eggs, and those which permit the eggs to hatch and the maggots to grow. Clean, dry sheep are not at all attractive to blowflies, and even those with a soiled, moist breech are not very attractive. However, scouring sheep, and more particularly sheep infested with maggots, are very attractive, at least to *Lucilia cuprina.* All sheep, including clean, dry animals which do not attract even *Lucilia cuprina,* supply an essential factor for attraction of the fly. This sheep factor must be associated with a putrefactive factor or odour before the sheep becomes sufficiently attractive for the fly to lay eggs on it. The putrefactive factor may be supplied by the sheep itself, for instance when it is scouring, or experimentally in the form of a cotton wool plug saturated with an attractive chemical which is tied in the fleece. Away from the sheep these materials have a much lower degree of attraction.

Bulletin No. 182.—" The Effectiveness of Various Mineral Dusts for the Control of Grain Pests," by J. S. Fitzgerald, M.Sc., Ph.D.

The protection of stored wheat from insect attack, investigated at the request of the Australian Wheat Board, has been one of the important wartime projects of the Council's Division of Economic Entomology. The problem was tackled in various ways, and one method of control studied was the use of " inert " mineral dusts. These dusts are not poisonous in the ordinary sense yet kill insects, apparently by their drying effect. Not very much was known of their action, but they offered the possibility of protecting wheat from weevil infestation, whereas other methods, such as fumigation, could only kill the weevils present at the time of treatment.

The dusts are mixed with the wheat and gradually either kill any weevils moving through the dusted grain or prevent their breeding. The protection lasts for several months at least, but the dust is readily removed before gristing the wheat. Conditions which influence the activity of the dusts are discussed in this Bulletin, but the main purpose is to assess the relative merits of various Australian mineral dusts.

The principal dust tested overseas was a finely ground crystalline silica, and a dust of a similar type has proved the most effective material tested in Australia. However, siliceous dusts cannot be used because of health hazards involved in handling treated wheat. The extremely fine state of division of those dusts is believed to contribute to their success.

Magnesite, dolomite, limonite, limestone, and hydrated lime, were among the dusts tested as substitutes. It was found that when ground to a dust mostly passing 200 mesh a dosage of 0·25 per cent. to 0·5 per cent. of the weight of the grain was usually sufficient to retard weevil development. Dusts mixed with grain of about 12 per cent. moisture content did not diminish in effectiveness even after two months. The lesser grain-borer, *Rhizopertha dominica,* was found to be more resistant to dusts than the rice weevil, *Calandra oryzae*, or the granary weevil, *Calandra granaria,* but even when the insects were not killed directly, their rate of breeding was greatly reduced. Though laboratory work only is described in the present publication, scale tests have been carried out in weevil-infested grain stores with very satisfactory results.

Forthcoming Publications of the Council.

At the present time, the following future publications of the Council are in the press :—

Bulletin No. 183.—" Experimental Determination of the Influence of the Red-legged Earth Mite *(Halotydeus destructor)* on a Subterranean Clover Pasture in Western Australia," by K. R. Norris, M.Sc.

Bulletin No. 184.—" Fellmongering Investigations," by F. G. Lennox, D.Sc., Margaret E. Maxwell, M.Sc., and W. J. Ellis, A.S.T.C.

Bulletin No. 185.—" Studies on the Mitchell Grass Pasture in South-Western Queensland. 2.—The Effect of Grazing on the Mitchell Grass Pasture," by R. Roe, B.Sc. (Agric.), and G. H. Allen, Dip. Agric. (Lawes).

Bulletin No. .—" Foundry Sand Resources of South Australia," by H. S. Cornelius and H. A. Stephens, B.Sc.

Bulletin No. .—" The General Ecological Characteristics of the Outbreak Areas and Outbreak Years of the Australian Plague Locust *(Chortoicetes terminifera* Walk.)," by K. H. L. Key, M.Sc., Ph.Dd.

PLATE 1.

A Survey of Houses Affected in the Beaumaris Fire. (See page 27.)

FIG. 1.—*House B3-2.*—Typical damage to completely gutted brick house.

FIG. 2.—*Rear of House A3-2.*—Brick house with cement rendering; Marseilles pattern tile roof. The house was showered with sparks and burning debris from other houses and from fiercely burning bushes on the hill immediately behind and to the north. Flames from the burning wood heap (the remains of which are shown) ignited the back verandah and outhouses from which flames and sparks swept into the roof under the tiles.

PLATE 2.

A Survey of Houses Affected in the Beaumaris Fire. (See page 27.)

FIG. 1.—*House A3-3.*—Douglas fir (oregon) frame house with Baltic pine weatherboards; corrugated iron roof; narrow eaves; all ventilators of pressed metal with small openings; louvres in the gable and covered by $\frac{1}{2}$-in. mesh wire netting. The house was sheltered to the north by House A3-4, which was slightly damaged, but a house immediately behind to the north-east was completely destroyed. The front and side hedges were burnt, the back fence destroyed, and the outhouses damaged. The house itself, although showered by sparks and burning debris from the adjacent burning house and scrub, was undamaged except for running and blistering of the paint.

FIG. 2.—*Rear of House A3-6.*—Brick veneer house with oregon and hard-wood framing; well constructed boxed-in eaves, the underside being lined with fibro-cement sheets; well fitting Marseilles pattern tile roof. Although showered with sparks, &c., from several surrounding houses the house was undamaged except for some blistering of the paintwork. Outhouses and the garage were destroyed, the only portion left standing being the front of the garage covered by fibro-cement sheets and the steel door of the garage.

PLATE 3.

A Survey of Houses Affected in the Beaumaris Fire. (See page 27.)

FIG. 1.—*Rear of House B2-2.*—Timber frame house with hardwood weatherboards; exposed rafter type eaves with lining boards on top of the rafters; Marseilles pattern tile roof. This house was surrounded by well-kept grounds with high trees on each side. The trees protected the house from the sparks and burning debris from the burning houses on each side, but the back fences and outhouses were destroyed.

FIG. 2.—*House B3-1.*—Douglas fir (oregon) framed house with Baltic pine weatherboards, corrugated iron roof, very narrow eaves; open-back vestibule covered with hessian blinds. The house was completely surrounded by dense tea-tree which, on the west side, touched the walls. The house was menaced by two fires: The first burnt the front hedge and the scrub on the east side; the second, about 30 minutes later, destroyed the outhouses, vegetation in the back yard, and the scrub on the west side. Although the fires burnt the scrub on all sides, the only damage to the house itself was the partial burning of a corner post.

PLATE 4.

A Survey of Houses Affected in the Beaumaris Fire. (See page 27.)

FIG. 1.—*House B3-4.*—Brick veneer house with Douglas fir and hardwood framing; boxed eaves; Marseilles pattern tile roof. The house next door was completely destroyed and the tea-tree hedge and trees between the two houses were burnt. Sparks, burning debris, &c., were prevented from entering through the high light over the door by a fly-wire screen behind the glass louvres. The only damage to the house was softening and flowing of the glass in the windows facing the burnt-out house.

FIG. 2.—*House B3-5.*—Brick house with flat " zinc-anneal " roof; ventilators scattered and screened with fly-wire. Although subjected to sweeping draughts of sparks from two burning houses across the road to the north-west the only damage to the house was the destruction of a canvas blind suspended from beneath the eaves on the east side.

PLATE 5.

A Survey of Houses Affected in the Beaumaris Fire. (See page 27.)

Fig. 1.—*House* B3-6.—Hardwood framed house with Baltic pine weatherboards; corrugated iron roof. The houses on the north side were undamaged but three houses immediately adjacent on the east and north-east were completely destroyed and sparks and burning debris enveloped the house. Apart from damage to the paintwork the house was not harmed. The $\frac{1}{2}$-in. mesh wire netting shown covering the eaves was observed to prevent burning particles entering under the roof. Also the netting prevented ingress of birds and rodents, thus keeping the space under the roof free from such highly inflammable matter as nests.

Fig. 2.—*House* B3-9.—This was a very solidly constructed timber frame house, with jarrah weatherboards and corrugated iron roof. Dense tea-tree and scrub grew close to the rear of house and the extensive grounds, which were heavily wooded with trees and shrubs, contained several outbuildings. The fire started at the back, the main fire sweeping under the house through large under-floor vents, which had been kept clear of vegetation. The house was completely destroyed in less than twenty minutes, and afterwards there was difficulty in finding the remains of the lighter timbers (4 in. x 2 in.) and only short lengths of 12-in. x 4 in. red gum (*E. rostrata*) bearers remained.

PLATE 6.

A Survey of Houses Affected in the Beaumaris Fire. (See page 27.)

Fig. 1.—*House B3-7.*—End view of the gable of the house (which was not damaged) showing the excellent spacing of the ventilators which are provided with fine mesh coverings.

Fig. 2.—Typical view of the rear of the four houses saved in Beach-road (section C1) showing the adjacent high trees preceded by grassland and scattered clumps of scrub.

PLATE 7.

The Relationship between Necrosis and Resistance to Virus Y in the Potato. (See page 48.)

Plant of potato hybrid 48 showing necrotic collapse to virus Y a month after inoculation. Inset shows inoculated leaf of 48 ten days after infection.

PLATE 8.

The Relationship between Necrosis and Resistance to
Virus Y in the Potato. (See page 48.)

Plant of potato hybrid 106 showing top necrosis to virus Y
a month after inoculation. Inset shows inoculated leaf of 106 ten
days after infection.